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# Presidential Address

## IRON ORE RESOURCES AND OPEN-CAST OPERATIONS IN THE MIDLAND OREFIELD

*By J. R. Menzies-Wilson, O.B.E.*

**I would like to say how very much I appreciate the honour of being your President for the coming year. I can assure you that I will do my best to further the interests of the Institute, and I am conscious of the fact that the standard set by your retiring President is a very high one.**

**In recent years, as a result of the growth in population and the increasing rate of development of civilization throughout the world, we are becoming conscious of the fact that the natural resources of the world are not unlimited, and may possibly be inadequate for our future needs. I have a special interest in iron ore and I feel that it would be appropriate for me to consider in this address some aspects of the production of iron ore, such as its probable availability and, in particular, our supplies of home ore and our methods of winning it from the ground. I know that in trying to reach any conclusion on availability I am treading on dangerous ground ; our knowledge of world mineral resources is limited, and anything that I say on this subject will necessarily be speculative. I feel, however, that we may be able to arrive at some broad conclusions in the light of such information as we now possess.**

### Introduction

From 1900, world production of crude steel has increased fivefold, until it is today about 150 million tons/year. In considering the future development of the steel industry certain factors must be taken into account. Steel may be replaced by other materials, and in fact it has already been superseded for certain uses. But, so far as can be foreseen, there will probably continue to be many purposes for which steel is particularly suitable. In addition, the quality of steel is being constantly improved and this has the effect of reducing the weight of steel required to do the same amount of work. Another point to be considered is the present unevenness in the production and consumption of steel throughout the world. If the less advanced countries continue to develop technically as quickly during the next fifty years as they have over the past fifty years, a greatly increased world demand for steel will be created, and this will, of course, affect the whole question of the supply of ore.

The volume of minerals so far extracted is an infinitesimal fraction of that which is believed to exist in the earth. In recent years, however, the rate of extraction has been rapidly increasing, and the amount won over the past fifty years greatly exceeds the total previously extracted. The real problem is not so much shortage of minerals as the fact that the rate of discovery of minerals may not keep pace with the rate of use.

This Address was presented at the Eighty-First Annual General Meeting of the Institute, on 26th April, 1950, with the retiring President, Sir Andrew McCance, F.R.S., in the Chair.

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### Ore Supplies

Most ore discoveries in the past have been outcrops or extensions of outcrops, but the stage has now been reached when almost all the more easily worked and accessible deposits of ore have been discovered and are being worked to a greater or lesser extent. The gradual exhaustion of these deposits will make it necessary to consider, in the near future, the obtaining of ore from more distant and less conveniently situated areas. It is possible to estimate approximately what quantities are available in other known deposits, but it is often difficult to obtain accurate information about the quality and the ultimate cost of extraction of the ore.

It is already evident that even in the more convenient areas the cost of extraction is likely, in time, to increase substantially. This refers, not to increases in price which accompany the fall in the value of money, which now seems to be inevitably associated with the passage of time, but to an increase in the real cost relative to other commodities. In the less accessible and convenient areas to be developed in the future, the cost will naturally be higher. In most cases there would be a large expenditure on railways and harbours, for example, and on the provision of necessary services which are not available locally. The actual cost of extraction may not be abnormal but the cost to the consumer is greatly increased by the cost of transport as, for example, in the newly discovered iron ore deposits in Labrador.

The work of exploration for hidden reserves, necessary to justify the opening up of mining operations in a new area, demands an outlay beyond the resources of most independent undertakings. There are, undoubtedly, still many small deposits which can be found and brought into use with a relatively small expenditure, but the discovery of new, large bodies of ore will ultimately become necessary, and will be a slow and expensive process.

### Discovery of New Deposits

Most countries are now aware of the need to carry out extensive geological surveys if they are to keep pace with the increasingly high demand for minerals. Improved exploration techniques, such as, for example, the aerial use of the magnetometer, will help to speed up the discovery of deposits as yet unknown; and the increasing cost of extracting ore from known deposits, combined with their gradual exhaustion, will provide the incentive for exploration.

The question whether a particular deposit discovered by survey shall be developed will be decided ultimately by economic considerations, taking into account the quality of the ore and its value to the iron producer. In very broad terms, the cost of converting the ore to molten iron comprises (1) extraction from the ground, (2) transport to the site where it is to be used, and (3) the actual smelting of the ore in the blast-furnaces and, in addition, of course, the requisite charges in each operation for depreciation of the plant and for maintenance.

Items (1) and (2) need little elaboration, but (3) is much more complex, depending as it does on the iron content of the ore, the reducibility of the ore to the requisite iron composition, and the need for the addition to the burden of silica or lime. A burden to which it is unnecessary to add any non-ferruginous material has a substantial advantage, other things being equal, over a burden to which substantial quantities of lime or silica have to be added, as these substances have to be melted at an additional cost without any return in iron.

It may add point to these remarks if it is stated that in some of the modern plants in this country the cost of coke manufactured on the site has quadrupled in the last 15 years owing almost entirely to the rise in price of coal.

## IRON ORE RESOURCES IN GREAT BRITAIN

The bulk of the ore in Great Britain is much lower in iron content than the ore which is available from abroad, but its relatively low cost, due to its accessibility, makes its use economic, particularly where it can form a self-fluxing burden. Furthermore, it is a commodity which is not affected in cost by external factors.

The iron ores in Britain occur generally in two forms: (1) Hematite, and (2) stratified ironstone in the Jurassic System (*see* Fig. 1). The existing known deposits of hematite are now small and lie in the western half of the country. Except for one mine in Glamorgan the only active workings at present are on the north-west coast, and as the supply is small, ore is obviously reserved for the making of special iron.

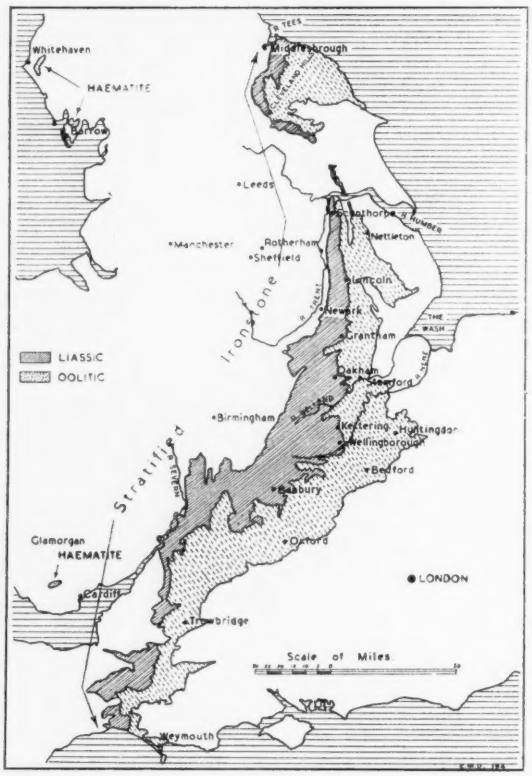


**Fig. 1—General location of iron ore deposits in England and Wales**

The stratified ironstone series runs from the River Tees southwards through Yorkshire, Lincolnshire, Nottinghamshire, Leicestershire, Rutland, Northamptonshire, Warwickshire, Oxfordshire, Wiltshire, and Gloucestershire, to the coast near Weymouth. The geological sequence of this system is shown in Fig. 2. There are three main beds of stratified ore; the Marlstone, the Northampton Sand, and the Frodingham Ironstone, but all three beds have the common characteristics of comparatively high phosphorus and comparatively low iron contents.

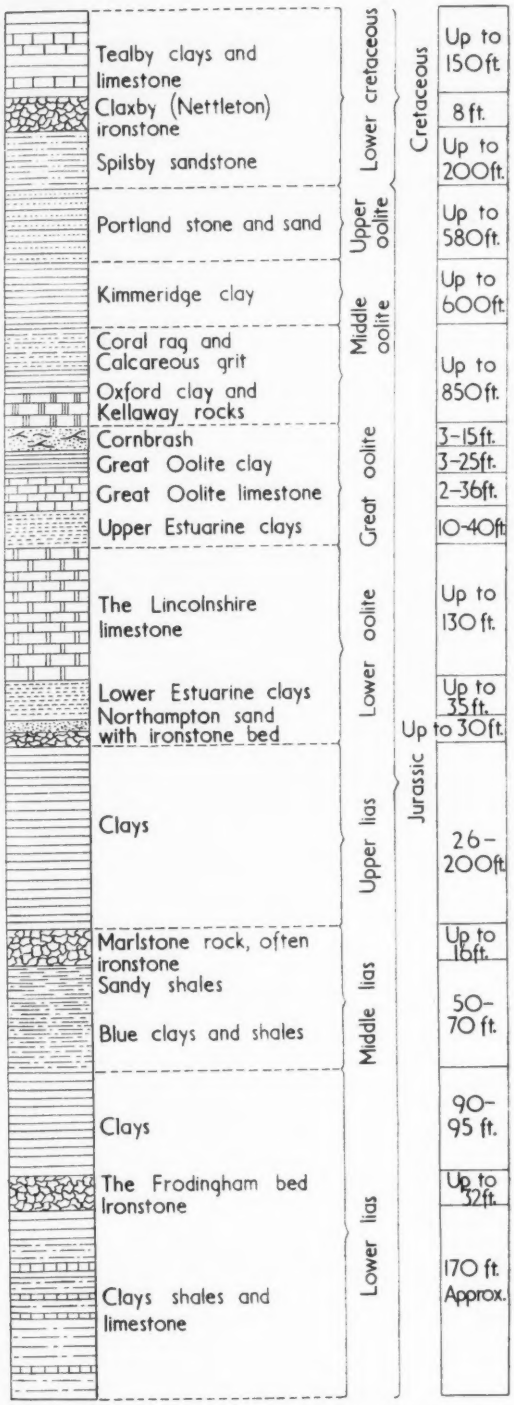


**Fig. 2—Section of Jurassic and Cretaceous strata, showing disposition of stratified ironstone deposits**

*Marlstones*—The marlstones are found in the Cleveland district and in two areas in the Midlands. The Cleveland district was the first major producing area in this country, production on a large scale being started in 1850, near Eston, but the output from this bed, which is now obtained entirely by underground mining, has since decreased, and to a large extent it has been replaced by the output from the Northampton Sand bed.

In the two Midland areas the ore has an iron content of 20–28%. The workable stone generally lies under shallow overburden consisting of clays and tractable material which is fairly easy to handle by modern methods.

*Frodingham Ironstone*—The Frodingham bed in North Lincolnshire is limy in character and has a low iron content of between 18 and 25%. For the sake of economy this ore is worked in the furnaces in conjunction with siliceous Northampton Sand Ironstone and foreign ore of high iron content. Owing to the sudden rise in the escarpment the future winning of this ore is almost certain to be underground and, indeed, some Companies have already started underground mines.

*Northampton Sand Ironstone*—The Northampton Sand Ironstone bed stretches from central Lincolnshire, across Leicestershire, Rutland, and Northamptonshire. The iron content of this ore is greater than that of the marlstone and the Frodingham ore, being 28–34% as quarried. As its name implies, it is on the whole a siliceous ore, but its composition varies greatly from area to area and within each area.

There is one further ore-bed, the Lower Cretaceous, but the reserves of this ore are limited and production is very small.

It is difficult to give even an approximate indication of the life of the reserves in the various fields because there are large areas of ironstone-bearing land where the quality and thickness of the ore bed have not yet been determined. There may be considerable areas where the ore may not be a workable proposition.

### Effect on Agriculture

The total area of agricultural land in England and Wales is approx. 24 million acres, of which approx. 200,000 acres are stratified ironstone-bearing land. Of these 200,000 acres it is estimated that 133,000 will be worked by underground mining, leaving 67,000 acres to be worked by open-cast operations, this last figure representing only 0·28% of the agricultural land. It should be understood that, under present methods, a considerable portion of this 67,000 acres, where the overburden makes it possible and economic, is being restored to agriculture and the balance is being planted with trees.

## NORTHAMPTON SAND IRONSTONE

This area provides more than half the production of home ore and is one of the most important of the nation's reserves, there being, excluding marginal lands, about 76,000 acres containing ironstone.

There is ample evidence that this ironstone bed has been worked from Roman times, and at the time of the Norman Conquest it vied with the Weald as one of the chief iron-producing centres in Great Britain. These conditions were maintained until early in the 18th century when, owing to the passing of very rigorous timber laws, preventing the cutting of timber for the making of charcoal, the production of iron in this area almost ceased for more than 100 years. It was not until the introduction of the railway system that the importance of this ironstone bed was realized and production began again, the ore being transported to the furnaces which had been erected in the coal-bearing areas of the Midland district. In the middle of the 19th century the first blast-furnaces were introduced on the ore bed itself, these being erected in 1857 at Weedon and Wellingborough. In 1913 the production of iron ore feeding the Midland blast-furnaces was 2 million tons. Subsequently there was a great change, caused partly by the decline of production in the Cleveland area, and partly by the increased use of this ore in the Midland area. An example of this was the erection in 1934 of an integrated works in Northamptonshire, relying entirely on ore supplies from the Northampton Sand Ironstone. Today, the production from this bed is nearly 7 million tons/year.

The chemical composition of this ore is extremely variable, chiefly in regard to lime and silica, but on balance it is siliceous. The iron content also varies, but to a lesser extent. The analysis of lime and silica in a single working face, particularly in the area south of the River Welland, can vary from chain to chain, and the ore can, in fact, turn from limy to siliceous, and *vice versa*, within 100 yd. of the working face. Further, as the whole face advances, sections that were siliceous may become limy in the advance of a few chains. Thus, when this ore alone is used, a long-term programme of ore winning requires very extensive exploration in front of the operations, and a very complicated programme of development, if the desired result of a self-fluxing burden to the blast-furnaces is to be achieved.

The early operations for winning this stone started in the outcrop along the valleys, working into the hillside. In many areas today it is no longer a question of working ore lying under shallow overburden ; in fact, underground mining is being developed, but at present the area involved is small.

Where the ore is to be worked by opencast methods in deep overburden the installation of large and expensive excavating machinery is entailed, and it is quite uneconomic to open the quarry and install machinery unless at least 10–12 years' work at maximum output is possible, to cover the initial expense.

Whereas it is the aim of the producer of ore for large integrated plants to supply an ore balanced in lime and silica, this is a result difficult to achieve today. It is therefore necessary to have available a number of quarries which can produce, at short notice, substantial quantities of siliceous or limy material to balance the ore mixture. Incidentally, owing to the local variation in quality, the efforts of the ore producers must be supported, in an integrated works, by an efficient method of mixing the varying ores from the different quarries, so that the resulting charge to the furnaces will be as consistent as possible, not only from day to day, but throughout each day.

It is generally agreed that this lean ore must be won and delivered at a low cost, which can be achieved only by extensive production, involving large capital commitments. Experience has shown that to obtain and handle that production efficiently in heavy overburden each quarry should provide approx. 400,000 tons of ore per year. If this amount is to be produced, there can be no time for delays of any kind, and any attempt to select suitable patches in a working face to suit the optimum burden is quite impracticable. When the quarry has been opened up, the removal of the overburden must progress continuously at such a speed as to keep ahead of the blasting and digging of the iron ore. The problem of maintaining a constant burden is emphasized by this need for a high rate of production in each quarry and, at the same time, the need to have sufficient quarries available to permit of a balanced mixture being obtained in spite of local variations in the analysis of the ore.

### Excavating Machinery

Where the Northampton Sand Ironstone lies near the surface, the overburden generally consists of lower estuarine clay, a material which could be handled, in the past, by primitive methods and, today, much more easily by modern machinery. No general limits to this type of working can be laid down since the lower estuarine clay varies in thickness from 4 to 35 ft. and, but for exceptional circumstances such as denudation by Glacial Drift (boulder clay), it is overlaid by the Lincolnshire limestone, which in the north-eastern part of the field reaches a maximum thickness of about 130 ft. This limestone varies in hardness, but generally is of such a nature that it requires preparation before being excavated. Consequently, in the deeper overburden the limestone must be adequately fragmented before it can be efficiently handled by excavators.

The development of excavating machinery over the last century has been governed by the gradual increase of overburden to be dealt with. In the past, when the producer was working at or near the outcrop of the bed, the overburden was removed by handshovel and barrow. In the same way, the ore bed was broken up and loaded by hand into small trucks for transport (*see* Fig. 3).

As the overburden became deeper, other and less primitive methods had to be found. In 1896 the first steam-crane navvy was introduced for loading ironstone, and a year later a similar machine, in conjunction with a transporter, was used for stripping overburden. Subsequently, the stripping of overburden was developed by the use of short-jib shovels, and later by long-jib shovels, which enabled the spoil to be dumped in the area from which the ore had

Fig. 3—Early ironstone workings in shallow overburden

Fig. 4—Electrically-operated face shovel, with 9-cu. yd. bucket, removing overburden up to 50 ft. in depth

Fig. 5—Small dragline baring ironstone in shallow overburden

Fig. 6—Walking dragline with 135-ft. boom and 3-cu. yd. bucket, capable of removing overburden up to 35 ft. in depth

Fig. 7—Large walking dragline, of American construction, with 215-ft. boom and 19-cu. yd. bucket

Fig. 8—Large walking dragline now under construction ; with 282-ft. boom and 20-cu. yd. bucket

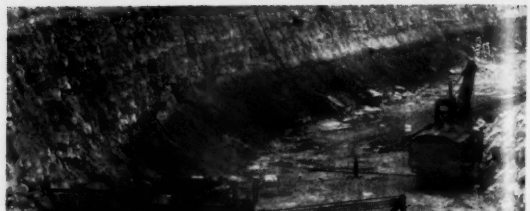

Fig. 9—Highwall horizontal drilling machine in operation. The view shows the drilling machine mounted on a platform, putting in blast holes at the base of the limestone in the highwall face. The drilling machine and platform are in turn supported on the jib and chassis of the caterpillar-mounted excavator, giving the desired degree of mobility to the unit

Fig. 10—Length of highwall face with horizontal blast holes charged and ready for firing. The strong limestone beds are self-evident

Fig. 11—The same length of face as in Fig. 10, after blasting, showing the highwall remaining *in situ* with little spillage

Fig. 12—Collapse of highwall, caused by overcharging and the omission of the buffer

already been extracted, thus overcoming the need for transporting the spoil on a separate machine.

In 1933 was introduced the first of the big electrically operated shovels built in this country, and since that time they have generally been used for stripping the ore when the overburden exceeded 30 ft. in depth (*see* Fig. 4).

The dragline was introduced in 1916 and in its first important application it replaced hand labour in the removal of shallow overburden where the power shovel was not suitable. The small dragline is the ideal machine for handling overburden up to 20–25 ft., and where there is no heavy limestone (*see* Fig. 5). A further development of this machine is one known as a walking dragline, the first of which was introduced into this country in 1941 (*see* Fig. 6).

Much larger walking draglines, developed in America, are now being introduced into this country. One of these machines has been in operation in opencast coal mining in the North of England for about a year, and one of the latest design, which can handle 70 ft. of burden in one operation, will be used on the opencast winning of ore within the next two months (*see* Fig. 7).

For some areas, where it has now become necessary to operate with an overburden greater than 70 ft., an even larger walking dragline has been designed, capable of handling up to 100 ft. of overburden. This is a British machine which will come into operation in September of this year. It has a boom length of 282 ft., a bucket capacity of 20 cu. yd., and it weighs 1600 tons. The boom of this machine is of an entirely new design, being constructed throughout of steel tubes. This saves over 10 tons in weight (*see* Fig. 8).

The development of excavating machinery has taken place almost entirely for the purpose of removing overburden, whereas the development of machinery for loading the ironstone has been confined to design and, generally, the changeover from steam to electricity has been made without any material

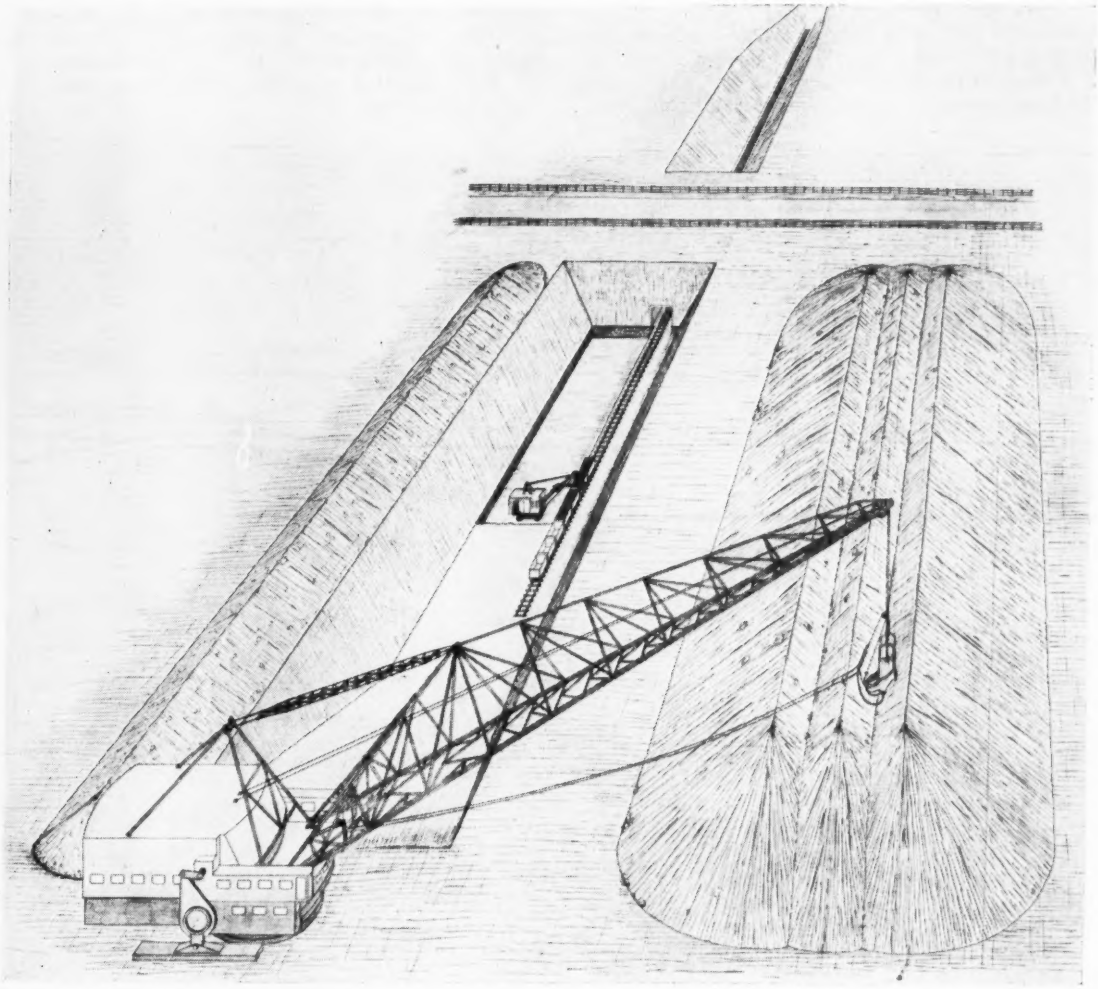

**Fig. 13—Method of opening-up a quarry in deep overburden by means of a large walking dragline**

**Fig. 14—Typical method of distribution of the explosive charge in horizontal blast holes. The width of the buffer fragmented by the previous series of blast holes is shown by *AB*, whereas the width of cut which is actually to be removed by the stripping machine is the 50-ft. width *AC*. The ground fragmented by the present series of holes is indicated by *BD*, of which the 30-ft. width *CD* forms the fragmented buffer for the next blast**

increase in the size of bucket. These machines have to load into railway wagons, trucks, or lorries, and it is the construction of these vehicles which limits the bucket capacity of the mineral-loading machines.

### Disposal of Overburden

The chief problems in winning Northampton Sand Ironstone from greater depths of overburden are connected with the preparation, handling, and disposal of the spoil, since in most cases a new area has now to be entered by opening up in moderately deep overburden, the major difficulty being the disposal of the spoil from the opening gullet. Conditions likely to be met in a new area today are an ore bed 7–10 ft. thick lying under from 50–70 ft. of overburden (*see* Fig. 13). For this operation a large walking dragline of the type already described would be used. This machine would itself dig the opening gullet, disposing of the spoil on both sides, with the larger proportion on the advance side of the quarry, resulting, of course, in subsequent double handling. The swell factor of some 30% also adds to the disposal problem.

The estuarine clay over the Northampton Sand Ironstone is generally overlaid, when the overburden is deeper, by a bed of limestone which varies in thickness from a few feet up to 130 ft., but in the opencast areas it rarely exceeds 30–35 ft. In some cases the bed consists of reasonably friable stone, except for a heavy band up to 5–6 ft. in thickness at the base. In other cases the whole bed consists of massive limestone.

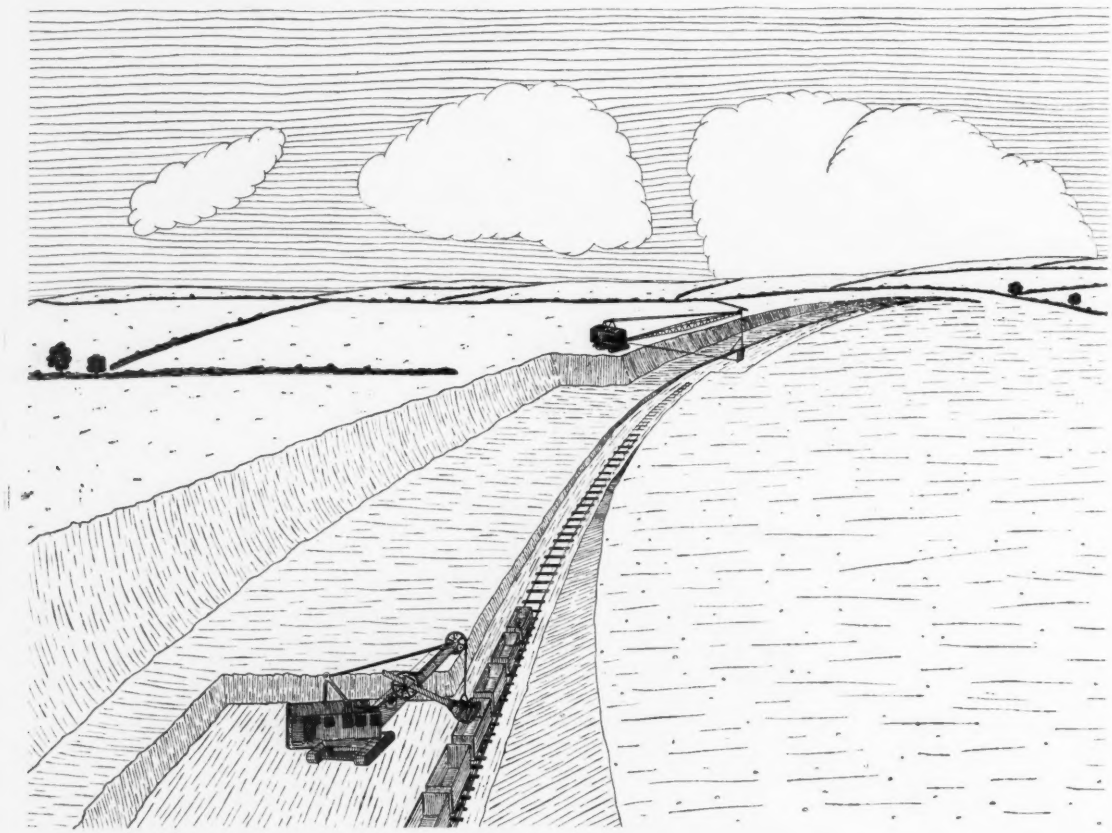

**Fig. 15—General method of operation using small dragline in shallow overburden. The shovel in the foreground is loading ironstone**

**Fragmentation**

One of the major problems of the producer today is the preparation of the limestone by fragmentation. There is no general rule on the correct method of treatment, as each area has to be considered individually.

Until 18 months ago the only machines used for the preparation of limestone for blasting were percussion and rotary-type vertical drills, but recently a new method has been introduced in this country, using a highwall horizontal auger drill, which makes a horizontal hole in the sand and clays at the base of the limestone. Owing to the jointing of the limestone and the fissures, it had not been possible, with vertical drilling, to perfect any method of fragmentation, and blocks weighing up to 15 tons and greater were left to be handled. Whilst it was possible, if difficult, to handle these blocks with a 9-cu. yd. shovel, the prospect of handling limestone of this size with a dragline presented even greater difficulties.

Some method of treatment was required to give better fragmentation of heavy limestone and at the same time so little displacement that the fragmented limestone would provide a reasonably level bed capable of carrying a machine weighing, say, 1600 tons, walking close to the edge of the highwall.

It was decided that the solution would probably be found by drilling the holes for the explosive charges horizontally in the sands and clays at the base of the limestone.

Experimental work, now developed to an operational scale, has proved this to be the case, as improved fragmentation without displacement of the limestone, and a consequent maintenance of the highwall, have been obtained. A horizontal drill was introduced, and for the sake of mobility and easy operation it was mounted on a small shovel-type machine. It was used to put in 6-in. dia. holes in the sands and clays below the limestone.

Under ordinary working conditions it is now possible to drill an 85-ft. hole in 35 min., as against a time of 10 hr. with a vertical churn drill. Figure 9 shows a highwall horizontal drilling machine in operation, whilst Figs. 10 and 11 show a highwall before and after a blast had taken place, very little limestone having fallen on to the benching of ironstone. This system of fragmentation is revolutionary so far as blasting operations are concerned, and experiments have disproved the earlier theory that a satisfactory fragmentation of limestone could be obtained only with the explosive in contact with the material to be fragmented.

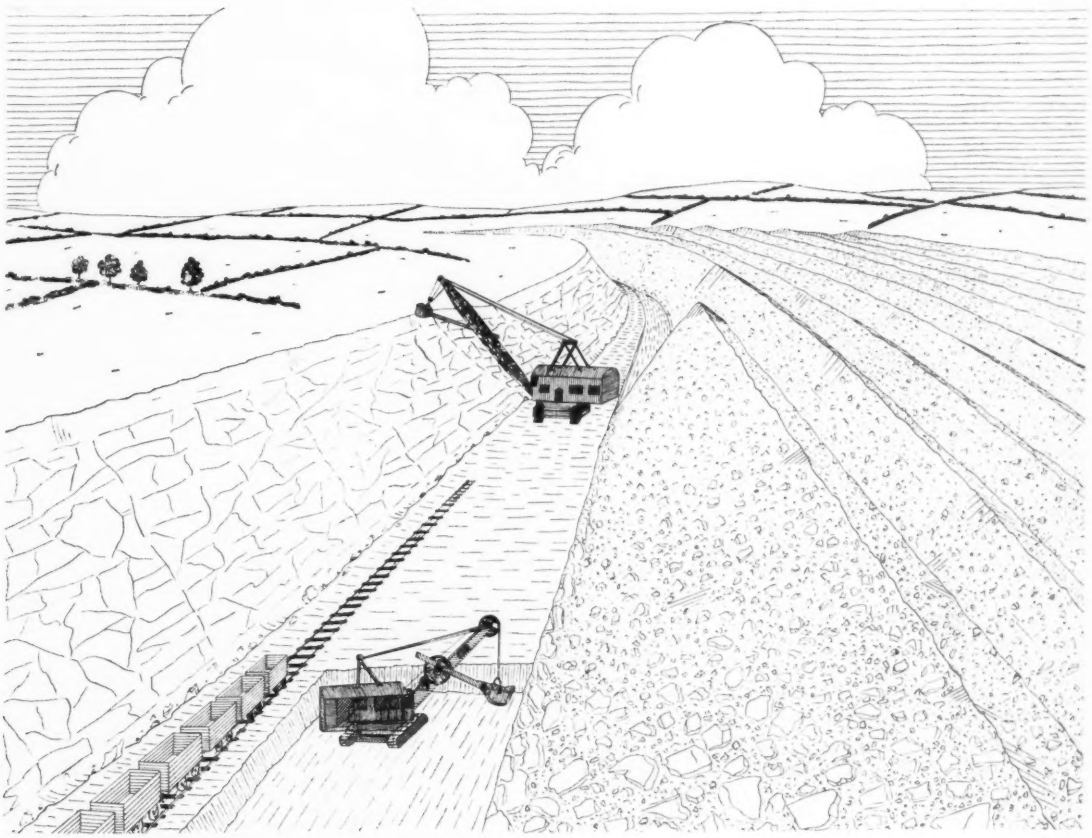

**Fig. 16—A 9-cu. yd. electric shovel in 50 ft. of overburden. In the foreground is the rock-loading machine**

A system known as buffer blasting has been developed to protect the highwall, and it is for this reason that the horizontal holes are placed at a depth of 80–85 ft. when the actual width of cut to be taken either by the shovel or the dragline is only about 50 ft. It will be seen from Fig. 14 that the whole of the explosive charge is placed in the back 50 ft. of the hole, the front 30 ft. being taken up by stemming. It is this development which has made possible the maintenance of stability. Figure 12 shows what happens to the highwall without buffer blasting and with a slight overcharge in explosive weight.

Although the experiments are not yet complete, sufficient evidence has been obtained to show that this method is correct for the conditions under which the operations are being carried out, and it appears that the proper charge per hole is approximately 1 lb. of explosive for every 5 cu. yd. of material.

There are obviously many advantages in operating drilling machinery of this type, the most important being that when the machine is operating on top of the ironstone it always has a firm base on which to stand, and, further, that it can follow up immediately behind the baring machine in putting in the next row of holes. It also has the advantage of making the servicing of the drilling machine fairly simple.

The remaining illustrations show what might be taken as reasonable methods of working under different conditions. Figure 15 shows the winning of ore in shallow overburden, where a small walking dragline, standing on the virgin surface, removes the overburden and leaves a level surface on the spoil side, whilst a power digger follows up and loads the fragmented ironstone into trucks.

Figure 16 shows an operation under different conditions with an overburden of about 50 ft., including a 30-ft. band of limestone. The method of operation is clearly shown in the diagram.

Finally, Fig. 17 depicts a method of working where overburden can be handled up to 100 ft. without the assistance of a secondary machine. It will be noticed that in order to achieve this the machine has to stand on the benching and dig above and below itself.

With our present knowledge the tendency will be for ore producers to introduce walking draglines in place of shovels when the opportunity presents itself. If, however, the overburden includes limestone of 50 ft. thickness, it will still be necessary to use the shovel excavator, and if, as is possible, the total

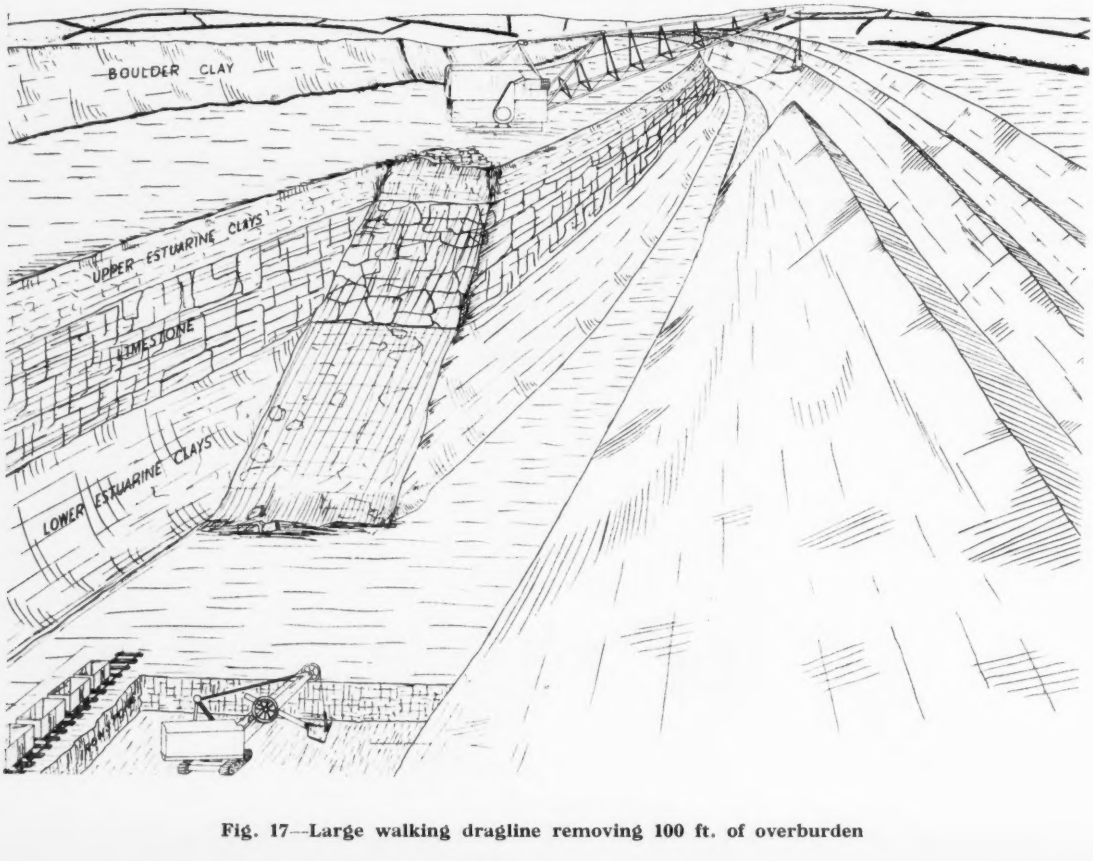


**Fig. 17—Large walking dragline removing 100 ft. of overburden**

overburden is something greater than 50–55 ft., a subsidiary operation will be necessary in conjunction with the shovel excavator.

## SUMMARY

An attempt has been made to indicate the main problems to be overcome in the future working of Northampton Sand Ironstone and, so far as Britain is concerned, it is almost certain that future developments in the production of ore will occur almost entirely in this bed. In common with other iron ores, the cost of producing home ore is bound to increase with its decreasing accessibility and the need for much greater capital expenditure on machinery and equipment.

Reference has been made to the increasing realization that the natural resources of the world are not unlimited. In regard to ironstone supplies the major problems of the future are : (1) the need to determine the location of hidden deposits, involving large-scale exploration, and (2) the ever-increasing cost of recovering less accessible deposits. Whilst the total supply, based on present consumption, is ample for a long time, it may soon become difficult to maintain the rate of discovery abreast of requirements. This problem will be intensified if the world production of steel is doubled in the next half century.

So far as this country is concerned, there is reasonably accurate knowledge of the reserves, but although the tonnages available are quite substantial they are not by any means unlimited. Prudence dictates that for the sake of succeeding generations, every effort should be made to utilize them wisely without waste, as they are an irreplaceable asset.

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# The System Gehlenite–Spinel

## RELATION TO THE QUATERNARY SYSTEM $CaO–MgO–Al_2O_3–SiO_2$

*By R. W. Nurse, M.Sc., F.Inst.P., and N. Stutterheim, B.Sc.(Eng.)*

The paper describes experimental work on the binary system gehlenite–spinel, and discusses its significance in the quaternary system $CaO–MgO–Al_2O_3–SiO_2$.

THE relation between the binary system gehlenite ($2CaO.Al_2O_3.SiO_2$) – spinel ($MgO.Al_2O_3$) and the quaternary system $CaO–MgO–Al_2O_3–SiO_2$ is shown in Fig. 1. The join between these two compounds was recognized by McCaffery[1] as being an Alkemade line,* although the experimental evidence on which he based his assumptions is not clear. From observations on the mineralogy of blast-furnace slags[3] (Fig. 2) it is clear that the combination spinel–melilite is a common one, even in slags of comparatively low MgO content; next to periclase, the spinel field appears to occupy the greatest volume within the quaternary system. The delineation of the spinel volume in this quaternary system would therefore be of great assistance in mapping some of the smaller primary phase volumes, and the present system is the first step towards the realization of this aim.

### EXPERIMENTAL METHODS

The quenching and thermal analysis methods already described by Lea and Parker,[4] and Nurse,[5] were used. Since the glasses formed are very stable and require long annealing periods before they become completely crystalline, it was necessary to ensure that each mixture was fully crystallized before starting heating curves, and in the quenching method the solidus and liquidus had to be approached from below, taking care not to overheat the mixtures. Failure to observe these precautions lead to anomalous results in the early work.

The analyses of the materials used are given in Table I.

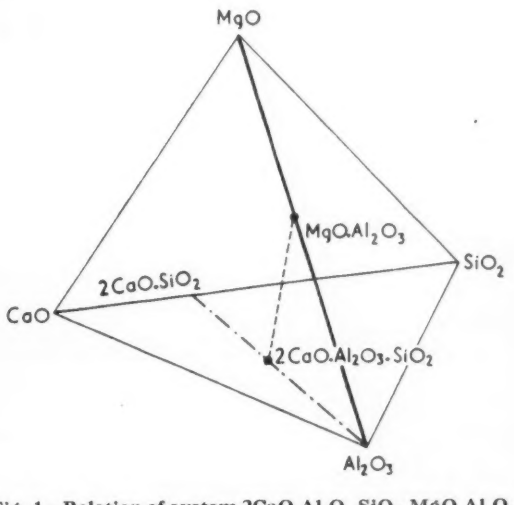


**Fig. 1—Relation of system $2CaO.Al_2O_3.SiO_2–MgO.Al_2O_3$ to quaternary $CaO–MgO–Al_2O_3–SiO_2$**

Manuscript received 22nd December, 1949.

Mr. Nurse is at the Building Research Station, Garston, England, and Mr. Stutterheim at the National Building Research Institute, Pretoria, South Africa.

* The terminology used is that recommended by Hall and Insley.[2] A join is the line drawn between any two primary phases; an Alkemade line is the join between two primary phases whose fields are adjoining.

## Table I

### ANALYSES OF STARTING MATERIALS

| Material | | CaO | MgO | $SiO_2$ | $Al_2O_3$ | (NaK)O | FeO | $Fe_2O_3$ | $TiO_2$ | $SO_3$ | Loss at 1200° C. | Purity,* % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $CaCO_3$ | | 56·16 | 0·01 | n.e. | 0·03 | 0·09 | n.e. | 0·01 | nil | 0·01 | 44·06† | 99·75 |
| MgO | | 0·07 | n.e. | 0·25 | 0·04 | n.e. | n.e. | 0·046 | <0·01 | 0·34 | 7·74 | 99·55 |
| $SiO_2$ | A | >0·005 | >0·002 | 99·82 | nil | 0·002 | n.e. | 0·007 | nil | nil | 0·036 | 99·82 |
| | B | n.e. | n.e. | 99·95 | n.e. | n.e. | n.e. | n.e. | n.e. | n.e. | nil | 99·95 |
| $Al_2O_3$ | | 0·044 | n.e. | 0·02 | n.e. | 0·234 | <0·001 | 0·026 | nil | 0·079 | 0·34 | 99·68 |

n.e. = not estimated. * Determined by difference, except for $SiO_2$. † Nil at 110° C.

Fig. 2—Blast-furnace slag, crossed polars. Isotropic spinel octahedra in melilite × 250

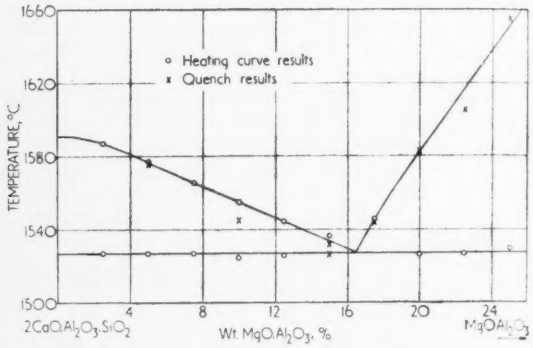


Fig. 3—System gehlenite–spinel

### EXPERIMENTAL DATA

Compositions, quench data and heating curve results are given in Table II.

The optical constants of gehlenite and spinel were identical with those given in the literature, and no evidence of solid solution was observed in either the optical or the thermal results. Figure 3 shows the constitution diagram constructed from the experimental data. The eutectic has a composition 83·5% gehlenite and 16·5% spinel (CaO 34·15%, MgO 4·70%, $Al_2O_3$ 42·85%, $SiO_2$ 18·30%) and is at 1527° C.

## Table II

### QUENCH DATA AND HEATING CURVE RESULTS

| Weight $MgO.Al_2O_3$, % | Temperature, ° C. | Quench Result | Heating Curve, ° C. Eutectic | Heating Curve, ° C. Final Melting |
|---|---|---|---|---|
| 2·5 | ... | ... | 1527 | 1587 |
| 5·0 | 1510 | $C_2AS$ and MA | 1527 | 1577 |
| | 1573 | $C_2AS$ and glass | | |
| | 1578 | Glass | | |
| 7·5 | ... | ... | 1527 | 1566 |
| 10·0 | 1543 | $C_2AS$ and glass | 1525 | 1555 |
| | 1548 | Glass | | |
| 12·5 | ... | ... | 1526 | 1545 |
| 15·0 | 1525 | $C_2AS$ and MA | | |
| | 1530 | $C_2AS$ and glass | ... | 1537 |
| | 1535 | Glass | | |
| 17·5 | 1540 | Glass and MA | ... | 1546 |
| | 1550 | Glass | | |
| 20·0 | 1520 | $C_2AS$ and MA | 1527 | ... |
| | 1535 | MA and glass | | |
| | 1580 | MA and glass | | |
| | 1585 | Glass | | |
| 22·5 | 1600 | MA and glass | 1527 | ... |
| | 1610 | Glass | | |
| 25·0 | 1500 | $C_2AS$ and MA | 1530 | ... |
| | 1650 | Glass and trace MA | | |

C = CaO ; A = $Al_2O_3$ ; S = $SiO_2$ ; M = MgO

### DISCUSSION OF RESULTS

The greater part of the system lies within the spinel primary phase field, and much of this region is beyond the temperature limits of the platinum/rhodium-wound furnace. The part of the system delineated does, however, establish an important point on the gehlenite–spinel boundary surface ; since the system is binary, this point will be a temperature maximum on the surface.

### Acknowledgments

The work described in this paper was carried out at the Building Research Station, Garston, Herts., England, and is published by permission of the Director of Building Research, Department of Scientific and Industrial Research, England, and the Council for Scientific and Industrial Research, Pretoria, South Africa. Analyses were made by F. J. McConnell and L. J. Larner.

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# Creep Testing by a Cantilever-Bending Method

*By G. T. Harris, M.A., F.Inst.P., and H. C. Child, B.Sc.*


## SYNOPSIS

Cantilever-bending creep testing is shown to have advantages over tensile testing for high temperatures and where the preparation of specimens must be reduced to a minimum, such as for unmachinable materials. There is good correlation between results obtained by this method and conventional testing at plastic strains up to 1·0%. For higher strains the results differ, owing partly to the constriction of tensile specimens under load, and tertiary creep is suppressed, although it can still be observed.


## Introduction

It is becoming increasingly necessary to design machines and structures to operate at temperatures above 800° C., and this has raised problems in creep testing alloys for use at these temperatures. The special problems involved are due partly to the high temperatures and partly to the fact that materials suitable for such use are often difficult or impossible to machine, so that a simple form of specimen is essential. Such materials may be produced as forgings, castings, or sintered products. A method is here described of creep testing in a cantilever bending machine which has certain advantages over other methods.

Tensile creep testing normally utilizes a screwed specimen, as a headed specimen introduces difficulties in the shackle design. It is not easy to make good threads either by precision casting or by powder metallurgy, and there are therefore good reasons for preferring a simpler test piece. The specimen for cantilever bending need only be a cylindrical or rectangular rod, preferably with a constriction at the centre, which is simple to form by casting or pressing, followed, if necessary, by grinding. The specimen for creep in compression is even simpler, but this method of creep testing has disadvantages.

To obtain a strain sensitivity of even $10^{-4}$ (0·01%) a tensile creep extensometer with 1-in. gauge length has to read to 0·0001 in. Compression creep units may use specimens of much smaller gauge length, necessitating even finer measurements. With a cantilever-bending method, however, it is quite easy to read the strain from the movement at the end of the beam with $10^{-4}$ strain producing a deflection of about $\frac{1}{32}$ in. This can be readily seen, and even subdivided, by eye. Creep rates of $10^{-6}$/hr. can thus be measured by using a scale divided in hundredths of an inch and read by means of a hand lens.

Both tension and compression creep methods have the disadvantage that the strain measurement is affected by temperature variations, so that a close control of temperature is necessary. Temperature variations, apart from their effect on the creep rate, do not affect the strain reading obtained in a cantilever test. Since accurate temperature control at very high temperatures is not easy, this is an additional advantage.

## PROCEDURE

The essential feature of the apparatus is that a small specimen is held in a block of heat-resisting steel by means of a slack push fit into a hole in the block. The specimen is loaded by means of a beam secured to the specimen in a similar way. Two side arms extending from the block carry a reference point against which the deflection of the beam is measured on a scale attached to the end of the beam (Figs. 1 and 2).

The original intention was to use a large muffle, open at both ends, and to have four specimens in each side of the block. Such a method was abandoned because of the difficulty of :

(1) Obtaining sufficiently close temperature control
(2) Eliminating temperature gradients which existed transversely across the furnace, and along the gauge lengths of eight specimens
(3) Replacing one specimen without disturbing the others or cooling the furnace
(4) Reaching high temperatures without excessive heat losses.

Accordingly, it was preferable to use an ordinary tube furnace with two frames back-to-back, the beams

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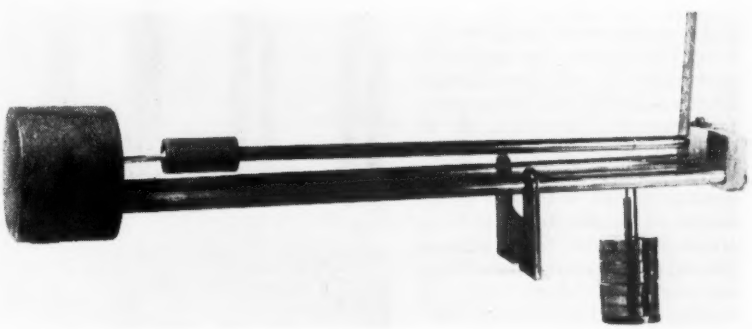

**Fig. 1– General view of cantilever creep testing unit without furnace**

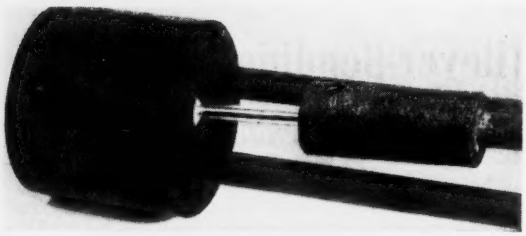

**Fig. 2—View of cantilever creep specimen, showing heat-resisting block and beam**

protruding from the two ends. These frames are clamped to the furnace, one at either end, by brackets and may be slid in and out to facilitate changing the specimen. By using shunt rheostats across three furnace zones good temperature uniformity is obtained between the two specimens and along the gauge lengths.

The specimen is stressed by a heat-resisting beam, 12 in. in length. The diameter of this beam is chosen so that the maximum fibre stress in the specimen caused by the moment due to the beam is low (0·5–6·0 tons/sq. in.). This ensures that the elastic limit of the material under test is not exceeded due to the dead weight of the beam. Further stressing is carried out by the addition of a carrier and slotted weights suspended from a groove at a fixed distance along the beam.

As large deflections result from small strains it is sufficiently accurate to measure the deflection by means of a scale graduated in hundredths of an inch. Such a scale may be read by eye with the aid of a lens to 0·005 in., representing a strain of about 0·003% with a specimen of 1-in. gauge length and 0·146 in. dia.

Figure 3 shows a selection of the types of specimen used. Specimen *A* is a cylindrical specimen, 0·146 in. dia. and with a 1-in. gauge length, which was used for most of this investigation. This type of specimen is the most suitable when the material is machinable. For higher-temperature work, where lower test stresses are required, specimens of greater gauge-length diameter have been used, for example, specimen *B* of 0·209 in. dia.

Specimen *C* is a rectangular specimen with a 0·8-in. gauge length of section 0·2 × 0·1 in., which is suitable for the direct pressing of powder-metallurgy compacts or which may be readily ground either from rectangular compacts or from other unmachinable material.

Specimens *D* and *E* have shorter gauge lengths, 0·25 in. and 0·062 in., respectively, to enable tests to be carried out to greater plastic strains. The limit with a specimen of 1-in. gauge length and 0·146 in. dia. is about 1·5%, but with specimen *E* it is possible to test many materials to fracture.

It should be noted that the bending moment varies along the gauge length if the specimen is of uniform diameter. The actual variation depends on the position of the centre of gravity of the beam and loading weights and is of the order of ± 5% of the applied moment at the centre section for most of the tests reported here on specimens of 1-in. gauge length.

Tests at high temperatures (*e.g.*, 1200° C.) require lower loads and much shorter loading beams, and here the error would be serious so that tapered specimens (Fig. 3*F*) are employed to equalize the stress along the gauge length.

## PREVIOUS WORK

Tapsell and Johnson[1] studied the bending of rectangular lead beams subjected to a uniform bending moment at room temperature. Their tests showed that the material behaved similarly in compression and in tension and that proportionality existed between strain and the distance from the neutral axis. Consequently, plane sections of the beam remained plane during the tests.

A graph of log-stress/log-creep-rate often shows a straight line at constant temperature ; the relation is given in equation (1), which implies that the creep rate varies as a power of the applied stress :

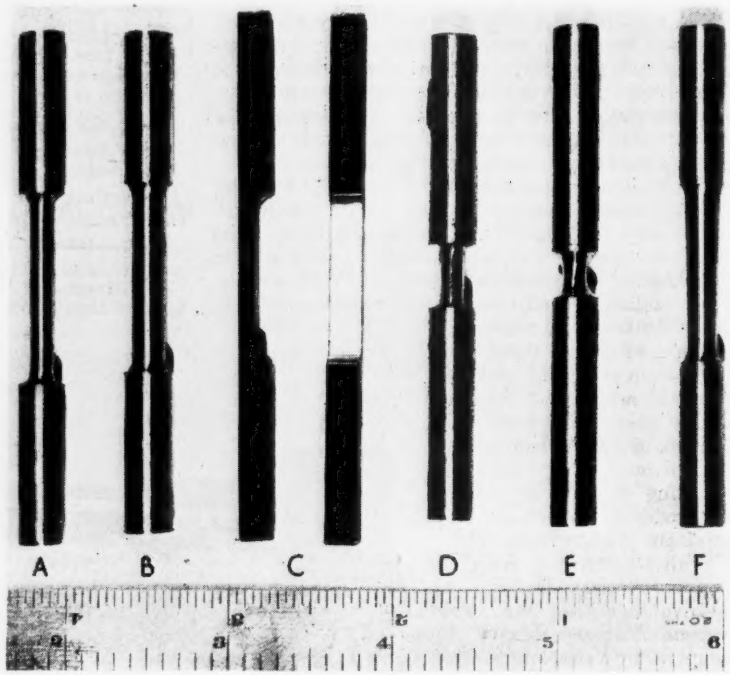


**Fig. 3—Cantilever creep specimens**

$$f = AR^n \quad \text{.....(1)}$$

where $f$ = stress
$R$ = creep rate
$A$ and $n$ = constants for the material at the test temperature.

Assuming that equation (1) holds and that for any particular fibre in the deflected beam the stress is the same as that which would cause the same strain rate in a tensile creep test, it can be shown (*see* the Appendix and also Tapsell[1]) that the stress in the outermost fibre ($f_A$) is related to the stress calculated for the elastic condition ($f_E$) by the following relation for a rectangular section :

$$f_A = \frac{n+2}{3} f_E \quad \text{.....(2)}$$

where $f_A$ = actual maximum fibre stress in plastically deformed material
$f_E$ = maximum fibre stress calculated from applied moment, using elastic theory of bending.

By means of a series of strain readings taken at different distances from the neutral axis, it was shown that in the experiments the redistribution of stress arising from creep was complete within 12 min. after initial loading and that the stress distribution then remained effectively constant.

MacCullough[2] has carried out similar work to that of Tapsell, but the bending and tensile creep are compared, assuming that :

$$f = B \log R + C \quad \text{.....(3)}$$

where $B$ and $C$ are constants for the material at a given temperature. MacCullough showed that there is good agreement between creep in compression and creep in tension for lead at room temperature.

Bolton[3] used cantilever loading of beams as a method of testing cast iron at 850° C., but no attempt was made to determine the actual stresses in the beam. His stress values are based on an assumption of the elastic theory. Popov[4] has recently discussed a method for calculating the stresses in rectangular beams in the early or transient stage of creep from known tensile data. The stress distribution calculated by this method is compared with that obtained using the same equation for steady creep as Tapsell and Johnson, and also using the hyperbolic sine creep law.[5]

## INTERPRETATION OF RESULTS

Before any results can be compared with tensile tests it is necessary to consider the stresses in a specimen deformed plastically. In the elastic condition the maximum fibre stress may be found from :

$$f_E = \frac{My}{I}$$

where $f_E$ = the stress in outermost fibre for elastic conditions
$y$ = the distance of outermost fibre from the neutral axis
$M$ = the applied bending moment
$I$ = the moment of inertia of the section of the specimen.

Once the elastic limit of the steel has been exceeded, the problem of estimating the value of the fibre stress becomes more difficult.

With the same assumptions as Tapsell, we can write :

$$f_A = K \cdot f_E$$

It can readily be shown that for rectangular beams the factor $K$ varies from 1 for perfectly elastic materials to $\frac{2}{3}$ for perfectly plastic materials. For circular beams this factor can be shown (*see* the Appendix) to vary from 1 to 0·588 under the same conditions. It is necessary therefore to assess the value of the factor $K$ in the creep tests conducted.

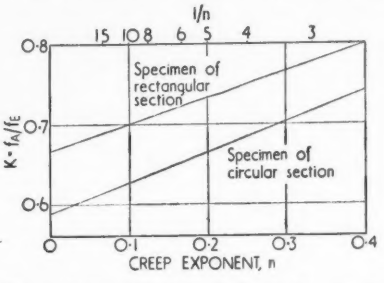


**Fig. 4—Dependence of the ratio $f_A/f_E$ on the creep exponent $n$**

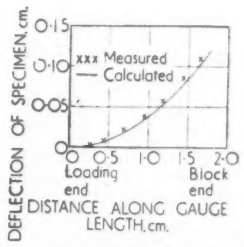


**Fig. 5—Comparison of calculated and observed beam deflections**

It is shown in the Appendix that if the following assumptions are made for a metal deforming by creep :

(*a*) Creep characteristics are similar in tension and compression
(*b*) $f = AR^n$

then the value of $K$ is related to the value of $n$, which can be obtained from the slope of log-stress/log-creep-rate graphs or, as pointed out by Tapsell,[6] from the slope of the log $R$/log $M$ line from the cantilever results themselves.

Figure 4 shows graphs correlating the values of $K$ and $n$ for circular and rectangular specimens.

From the slopes of the log-stress/log-minimum-creep-rate plots for two austenitic alloys, *G18B* and *G32*, the values of $n$ over a wide temperature range lie within 0·075 and 0·15 (*i.e.*, the creep rate varies as approximately the tenth power of the stress). The corresponding values of $K$ are then in the ranges 0·69–0·72 and 0·62–0·65 for rectangular and circular specimens respectively. Assuming values of 0·70 and 0·63 respectively for these sections will introduce a stress error of not more than ± 3% and will enable the effective fibre stress to be calculated from the loading of the beam.

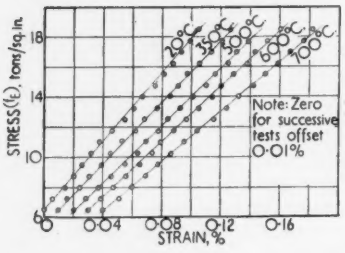


Fig. 6—Load–deflection tests on *G18B* at 20–700° C.

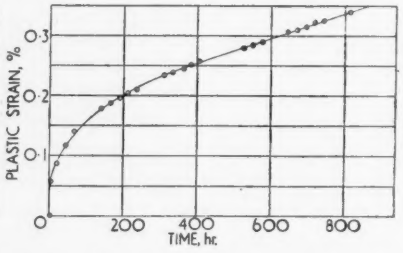


Fig. 7—Typical cantilever creep test ; $f_A$ = 12·3 tons/sq. in., temperature 750° C., cobalt-base alloy

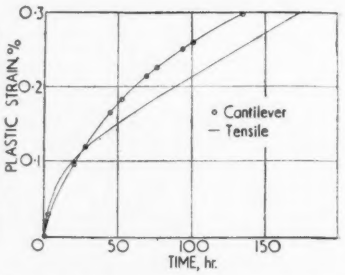


Fig. 8—Comparison of cantilever and tensile creep curves at $f_A$ = 8·5 tons/sq. in., temperature 700° C., alloy *G18B*

It should be emphasized that these values will hold only after some time has elapsed and the specimen has settled down to a period of steady creep.

The correlation of fibre strain and beam deflection is made on the assumption that the specimen bends to a circular arc. Figure 5 shows for an actual test the comparison between the deflections observed along the specimen and the curve derived from the beam deflection, assuming bending to a circular arc. There is seen to be adequate agreement. There is slightly greater curvature at the end of the test piece remote from the loading beam due to the gradient of the bending moment along the specimen, but the error introduced is in most cases negligible. Where it cannot be neglected tapered specimens have been substituted.

## TESTS IN THE ELASTIC RANGE

A series of load–deflection tests was carried out on *G18B* at various temperatures. To produce a sufficiently high elastic limit the specimens were first loaded to give 1% plastic strain and unloaded. The subsequent reloading curves were used to estimate the elastic modulus. The results are shown in Fig. 6, and in the following table the values obtained are compared with those determined by orthodox methods :

*Temperature Variation of Young's Modulus for G18B*

| Temperature, °C. | Young's Modulus, tons/sq. in. (Cantilever Apparatus) | Percentage of True Value |
|---|---|---|
| 20 | 11,500 | 85 |
| 350 | 10,300 | 84 |
| 500 | 9710 | 85 |
| 600 | 9140 | 86 |
| 700 | 8280 | 84 |

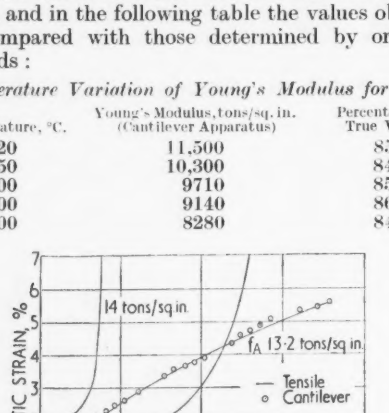


Fig. 9—Comparison of cantilever and tensile creep curves ; $f_A$ = 13·2 tons/sq. in., temperature 700° C., alloy *G18B*

It will be seen that the experimental points lie reasonably on straight lines, but that the elastic modulus values are consistently low by about 15% because of additional deflection due to the deformation of the beam and the block. For tests in the plastic and creep range the proportion of the deflection due to the deformation of the beam and block is much lower, probably leading to errors in strain estimation of the order of 3%. This is because under such conditions strain is very steeply dependent on stress (contrast elastic straining) and the loading of beam and block is only a fraction of that in the specimen itself.

## CREEP TESTS

The results of a typical creep test, using this apparatus, are shown in Fig. 7, which records a strain/time curve with a creep rate at 800 hr. of about $2{\cdot}1 \times 10^{-6}$ per hr. The deviations of the points from the smooth curve are in no case greater than 0·004% strain, which corresponds to slightly less than $\frac{1}{100}$-in. beam deflection.

Figures 8 and 9 show comparisons between tests on *G18B* at 700° C., using cantilever and conventional tensile creep-testing equipment. At a low stress and creep rate (Fig. 8) the agreement between the two tests is good, and whilst 0·3% strain is reached somewhat sooner by the cantilever test, the creep rate at this strain is the same for both the tensile and the cantilever tests. At higher stresses and creep rates (Fig. 9) the agreement is good up to 1% strain, but at higher strains the two curves differ. This is due partly to the increase in stress in the tensile test caused by a

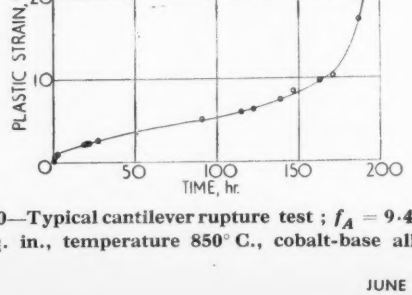


Fig. 10—Typical cantilever rupture test ; $f_A$ = 9·4 tons/sq. in., temperature 850° C., cobalt-base alloy

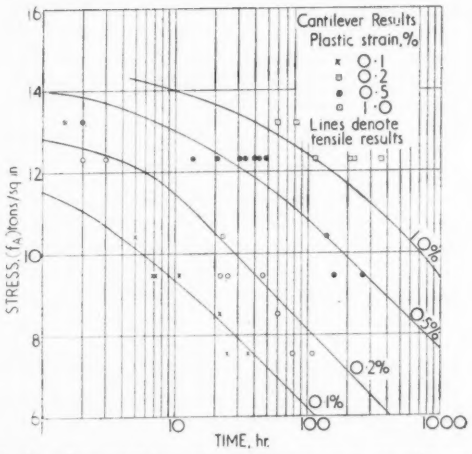


**Fig. 11—Comparison of cantilever and tensile creep tests on *G*18*B* at 700° C.**

reduction in cross-section, and partly to a gradual decrease of stress in the cantilever test as the stress distribution approaches its steady state during the second stage of creep. Whereas, here, the third stage of creep has been suppressed in the case of the cantilever specimen, it is worthy of note that tertiary creep can be obtained in bending (Fig. 10), corroborating the work of Sully,[7] who has noted the existence of tertiary creep in compression.

Further comparisons of cantilever and tensile creep are shown in Figs. 11 and 12, which again refer to *G*18*B* at 700° C. In these it should be carefully noted that the lines are the values obtained from a series of tensile creep tests carried out on similar material not necessarily from the same cast. The agreement for 0·1% and 0·2% strains is excellent, and it is good for 0·5% strain; above this value deviations occur due to the factors mentioned which accelerate the creep in the tensile test and lower the creep rate in cantilever tests and alter the time taken

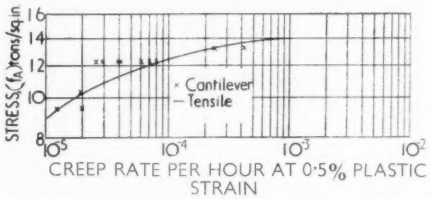


**Fig. 12—Comparison of cantilever and tensile creep tests on *G*18*B* at 700° C.**

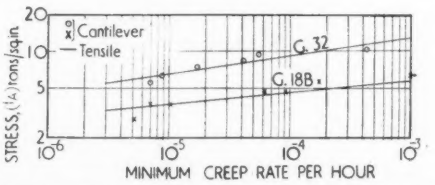


**Fig. 13—Comparison of cantilever and tensile creep tests of *G*18*B* and *G*32. Minimum creep rate per hour**

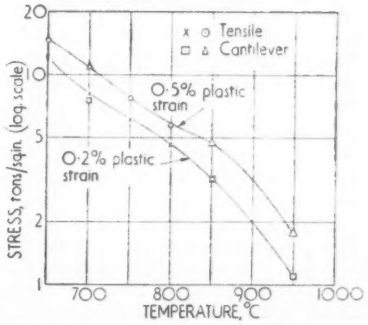


**Fig. 14—Creep strength of *G*18*B* at 650–950° C. ; cantilever and tensile tests. Indicated plastic strain in 100 hr.**

to reach, for example, 1% strain. For the same reason the graph (Fig. 12) of creep rate at 0·5% plastic strain shows fair agreement, but there is less satisfactory correlation between the plots of creep rate at 100 hr. Incidentally, both Figs. 11 and 12 show the scatter obtained in testing, by the range of values recorded at a stress of 12·3 tons/sq. in.

Having established by the above tests the possibilities of the method, Fig. 13 shows the results of a series of tests comparing at 850° C. the strengths of *G*18*B* and *G*32. Here, again, the lines are the mean results of tensile creep tests and the points refer to cantilever creep. The ratio of strengths of the two alloys found in this way agrees well with that obtained by conventional methods.

At this higher testing temperature the correlation between tensile and cantilever plots of minimum creep rate is quite satisfactory. At still higher temperatures than this the method is of increasing value owing to the difficulties in tensile testing at such temperatures. Figure 14 shows the creep strength of *G*18*B* plotted against temperatures between 650° and 950° C., some of the points being from tensile and some from cantilever tests.

Tests have been carried out at the still higher temperature of 1100° C. on *R*23, an austenitic 25/20 alloy. A modified apparatus, with a much shorter loading beam and tapered test specimens, was used. The results are incorporated in Fig. 15, which shows the effect of temperature on the creep strength of *R*23, some points being the results of tensile tests and others of cantilever tests.

Both Fig. 14 and Fig. 15 illustrate the general applicability of this method of testing.

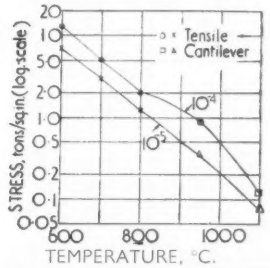


**Fig. 15—Creep strength of *R*23 at 600–1100° C. ; cantilever and tensile tests. Minimum creep rate per hour**

## CONCLUSIONS

A number of tests have been carried out on heat-resisting steels to correlate creep in cantilever bending with conventional tensile creep tests. Theory shows that if the stress can be related to the creep rate by a power law with constant exponent, a simple relation can be derived to correlate the two types of test. The relation is that the maximum fibre stress in the bending creep specimen, calculated by simple elastic theory, must be multiplied by a factor $K$, and the resulting stress will produce similar creep deformation in tensile tests. For three heat-resisting austenitic alloys investigated the factors are 0·70 and 0·63 for specimens of rectangular and circular cross-section respectively. These factors are obtained from the results of tensile creep tests, and should not be in error by more than $\pm$ 3%. Actual correlation between cantilever and tensile tests on these alloys, using these factors, showed good agreement within the limits of experimental error. Good correlation is obtained for strains under 1%. For higher strains the shapes of the creep curves differ, but tertiary creep does occur in bending.

The simplicity of the apparatus and method makes it suitable for tests on unmachinable alloys, precision castings, or sintered products, and for testing at temperatures of 1100° C. or higher, where the ordinary methods involve great practical difficulties. For testing new materials (*e.g.*, sintered products), which may not obey the criterion $f = AR^n$, the test is still of value as a means of comparing these materials, assuming either elastic conditions or an arbitrary factor $K$.


## Acknowledgments

The authors' thanks are due to Mr. D. A. Oliver, Director of Research, The B.S.A. Group of Companies, for interest and encouragement in the work described, and to Mr. D. Tattersfield for assistance in solving the integral referred to in the Appendix.


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## APPENDIX

Tapsell and Johnson[1] have shown that, for rectangular specimens :

$$f_A = \frac{n+2}{3} \times f_E$$

where $f_A$ = maximum fibre stress in plastically deformed materials
$f_E$ = maximum fibre stress calculated from the applied moment using the elastic theory of bending
$n$ = constant for material at a given temperature which can be obtained from tensile creep test using the formula

$$f = AR^n$$

where $f$ = stress
$R$ = creep rate
$A$ = a constant for the material at a test temperature.

Consider a circular specimen of diameter $d$ subject to an applied moment $M$. When this specimen is strained in the plastic region, the applied moment

$$M = \frac{\pi f_E d^3}{32}$$

must also equal the integral of the moments of the actual fibre stresses about the neutral axis ; that is :

$$M = 2\int_0^{d/2} 2.f.y \sqrt{((d/2)^2 - y^2)}.dy$$

Now $R = ky$, where $k$ = a constant (*i.e.*, rate of strain in any fibre is proportional to its distance from the neutral axis). Then $f = k_1 y^n$, where $k_1$ is another constant. Hence :

$$M = 4\int_0^{d/2} k_1 y^{n+1} \sqrt{((d/2)^2 - y^2)}.dy$$

This integral can be solved by the use of gamma functions ; whence :

$$M = 2k_1 \left(\frac{d}{2}\right)^{n+3} \sqrt{\pi} \left(\frac{\Gamma\frac{(n+2)}{2}}{\Gamma\frac{(n+3)}{2}} - \frac{\Gamma\frac{(n+4)}{2}}{\Gamma\frac{(n+5)}{2}}\right)$$

Now

$$f_A = k_1 \left(\frac{d}{2}\right)^n$$

$$\therefore M = \frac{\pi f_E d^3}{32} = 2f_A \left(\frac{d}{2}\right)^3 \sqrt{\pi} \left(\frac{\Gamma\frac{(n+2)}{2}}{\Gamma\frac{(n+3)}{2}} - \frac{\Gamma\frac{(n+4)}{2}}{\Gamma\frac{(n+5)}{2}}\right)$$

$$\therefore f_A = f_E \times \frac{\sqrt{\pi}}{8\left(\frac{\Gamma\frac{(n+2)}{2}}{\Gamma\frac{(n+3)}{2}} - \frac{\Gamma\frac{(n+4)}{2}}{\Gamma\frac{(n+5)}{2}}\right)}$$

But

$$f_A = K.f_E$$

$$\therefore K = \frac{\sqrt{\pi}}{8\left(\frac{\Gamma\frac{(n+2)}{2}}{\Gamma\frac{(n+3)}{2}} - \frac{\Gamma\frac{(n+4)}{2}}{\Gamma\frac{(n+5)}{2}}\right)}$$

The values of $K$ for corresponding values of $n$ may be evaluated from tables of gamma functions. For example, when $n = 0$, *i.e.*, for perfectly plastic material, $K = 0{\cdot}5882$. Figure 4 shows the variation of $n$ with $K$, and it will be seen that over the range of values of $n$ under consideration, there is an approximately linear relationship.

# The Effect of Cold-Work on Steel

*By J. H. Andrew, D.Sc., and H. Lee, Ph.D.*

## FOREWORD

The effect of cold-work on metals and alloys has been studied by different workers over many years, and some of the basic principles and facts are well known. For example, plastic deformation takes place in the form of slip by shear, the planes and directions of slip being dependent primarily on the lattice structure of the metal. Deformation gives rise to lattice distortion and grain fragmentation, along with other changes in the properties, notably an increase in hardness and strength. The study of these fundamental principles has led to many theories, amongst which the more recent and perhaps the most attractive one is the theory of dislocation proposed separately by Taylor,[1] Polanyi,[2] and Orowan.[3] In this theory the importance of lattice imperfections and local strain is emphasized, and the process of slip is identified with the propagation of dislocations in the metal crystals or grains. As the degree of cold-work increases, the density of such dislocations increases likewise, but tends to approach a limiting value. On the assumption that work-hardening is directly associated with the density of dislocations, this theory has been used satisfactorily to explain the change in properties of cold-worked metals and alloys, if they are homogeneous and their work-hardening characteristics obey the normal shear-stress/strain criterion. Even so, there seem to be slightly divergent opinions concerning the framework of this theory. For instance, Taylor's mathematical treatment deals mainly with stored energy in terms of lattice distortion, whereas Bragg's calculation[4] is based chiefly on the limiting size of the crystallites. Nevertheless, such divergencies may be relatively unimportant, because the real difference between these two schools of thought is purely a difference in the 'path' which they have taken. The fundamental assumptions and the final conclusions of the two methods are essentially the same.[5]

The theory outlined above is not expected to apply to a heterogeneous material. Indeed, with certain heterogeneous alloys, the stress/strain relationship may, when deformation proceeds to a certain stage, depart from its normal criterion; steel is known to be in this category. Thus, the work-hardening behaviour of carbon steels is known to be different from that of pure iron in many respects. With pure iron, the hardness of the specimen increases most markedly at the beginning of deformation; after this, the rate of increase becomes progressively smaller until the specimen approaches a constant value in hardness. The general characteristics of the work-hardening curve in this case are similar to those of other metals and can be predicted by the dislocation theory. In steel, however, there is an abrupt increase[6] in the strength of the material at about 50–80% reduction by cold-drawing, and the degree of deformation corresponding to this latter increase varies systematically with the carbon content (0·3–0·77%) of the steel. Moreover, cold-worked steels have invariably shown[7] an increase in hardness or strength after tempering at low temperatures, the maximum effect usually occurring on tempering at about 300° C. There are indications that this secondary hardening increases with the carbon content of the steel and is generally more marked if the degree of cold-work initially received by the specimen is greater. 'Secondary' hardening has not been observed to any appreciable extent with cold-worked pure iron specimens.

When these differences in the work-hardening properties of iron and carbon steels are considered, it seems clear that cold-work on steel may involve other changes in addition to those which can be explained in terms of the normal mechanism of plastic deformation. Since the most obvious difference between iron and steel lies in the presence of carbon, it was thought that the many 'unusual' features associated with the work-hardening properties of steel may be considered from a metallurgical point of view. It was with this in mind that the authors, in an earlier paper,[8] drew attention to certain features and compared them with the effect of quenching. They advanced the hypothesis that austenite was formed in steel during cold-working.

In the discussion on that paper, the austenite theory was not generally accepted as proved for normal forms of cold-working. There are indications, however, that with dynamic loading where the deformation is extremely rapid and localized, or in the case of working which involves intense heat due to surface friction, the formation of austenite and martensite does occur. At the same time, the various effects of internal strains upon physical and mechanical properties of steel are not sufficiently realized. It was with this in mind that the present work was undertaken, experiments being carried out on a much more extensive scale and covering both normal and rapid rates of cold-working. As a result, the effect of internal strains in ferrite and in cementite upon some of the properties of steel can be differentiated, and it appears that austenite formation in steel is restricted to special forms of cold-working involving either dynamic loading or intense surface friction. In presenting these results, it is hoped that some of the misconceptions in the earlier paper will be rectified.

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Professor J. H. Andrew and Dr. H. Lee are at the University of Sheffield. The co-authors of the following Sections were all at the University of Sheffield when these investigations were carried out.

No attempt is made to review the many publications on this subject, since the bibliography is very extensive and incomplete abstracts could lead only to misunderstandings.

The experiments described were carried out by a number of independent workers, and for this reason the paper is subdivided into Sections in accordance with the investigators concerned.

A number of commercial steels were used in this investigation, and their compositions are listed in Table I. Some of these analyses are incomplete, but it may be helpful to point out that steels *A* to *F* all belong to the plain carbon category, and the chief interest is therefore the respective carbon contents.

With the exception of the experiments described in Sections IV and V, cold-working in the form of wire-drawing was employed throughout. The drawing operation was carried out on a drawing bench fully mechanized and fitted with steel dogs and rotating drums driven by a motor coupled with gears. The drawing speed was approximately 5 ft./min. and dry soap was the only lubricant used. The ' blank rods,' before drawing, were machined to sizes usually between 0·2 and 0·3 in., although sizes up to 1 in. dia. were used when experimental conditions demanded. Tungsten carbide dies were employed, and the die angles varied from 6 to 8° for 1-in. and 12 to 14° for $\frac{1}{4}$-in. holes. Drawing was carried out in steps of $\frac{1}{32}$ in. for $\frac{1}{2}$ to $\frac{1}{4}$-in. dia. bars, although for small wires the reduction per draft was considerably less. To avoid excessive heating during drawing, a jet of cold water was played on to the specimen where it left the drawing die.

The cold-working described in Section V was carried out by compression and that in Section IV by means of a dynamic load impact which reduces the height of the specimen. The various procedures and some of the steels employed which are not given in Table I will be described in the two Sections concerned.

## Section I—EFFECT OF CARBON ON THE WORK-HARDENING PROPERTIES OF STEEL

*With P. L. Chang, B.Sc., Ph.D., B. Fang, B.Eng., Ph.D., and R. Guenot, E.C.P.(Ing.), Ph.D.*


### SYNOPSIS

Hardness, dilatation, density, and thermo-magnetic measurements have been carried out on cold-drawn specimens of a series of plain carbon and certain alloy steels. The difference in the work-hardening properties between high-carbon steels and low-carbon iron is marked. Unlike low-carbon iron, high-carbon steels show a rapid increase in hardness on cold-drawing towards high reductions and, at the early stages of cold-working, the shape of the work-hardening curve is shown to vary with the initial treatment given to the specimens. The results also confirm those of earlier workers, that with cold-worked high-carbon steels there is a marked increase in hardness on tempering up to 300° C. ; such a secondary hardening is not evident in cold-worked iron.

Several changes have been observed from dilatation measurements in the longitudinal direction on cold-drawn steel wires, the most marked being an expansion between 150° and 450° C. This change is mainly directional, because the increase in length is accompanied by a contraction in diameter. The change is more pronounced if the carbon content is higher and, with a given steel, when the initial structure is pearlitic rather than sorbitic.

The density of steel decreases on cold-working. With cold-drawn specimens, the maximum decrease is of the order of 0·2–0·3%. On subsequent tempering, the recovery of density takes place mainly between 400° and 700° C. With ingot iron, however, the decrease in density on cold-working and the extent of recovery on subsequent tempering appear to vary with the amount of inclusions present.

Thermo-magnetic experiments have been carried out on cold-worked steels. With a 0·7% C steel, cold-drawn beyond about 50% reducton, the cementite Curie point is markedly affected. At 90% or higher reductions, the thermo-magnetic curve is perfectly smooth, showing no discontinuity corresponding to the normal cementite Curie point. The magnetic saturation of the steel at room temperature, however, is apparently unchanged after cold-working. The absence of the normal cementite Curie point has also been observed with tempered martensite.

X-ray examination on a cold-drawn high-carbon steel shows that the intensity of cementite lines is appreciably weakened when the drawing reductions exceed 50%. Micrographic observations confirm that cold-worked steels are prone to graphitization on sub-critical annealing.


THE experiments described in this Section are mainly concerned with the effect of carbon content and treatment upon certain physical and mechanical properties of cold-drawn steel wires. Owing to the small and varying size of these wires, hardness determinations were used in place of tensile tests for mechanical properties. The bulk of the work was on plain carbon steels, although results from some low-alloy steels are included.

### HARDNESS EXPERIMENTS

All hardness measurements were made with the Vickers diamond pyramid machine. A number of impressions were made on a polished transverse section at regular intervals along the diameter of the wire, and the average value was taken to give the representative hardness figure. Since, in hard-drawn wires, the fibre structure was very pronounced, tests were also carried out along the longitudinal axis. There was very little difference between the results obtained in the two directions.

The results are dealt with in two parts : hardness values obtained on as-drawn wires are plotted against the percentage of reduction in cross-section by drawing, and those for tempered wires are plotted against the tempering temperature.

**As-Drawn Wires**

*Plain Carbon Steels*—Hardness curves from a series of plain carbon steels after various degrees of cold-drawing are given in Fig. 1*a*. Although the values of the five steels were different, because of the different carbon contents, they all show similar characteristics in the early stages of cold-work, below approximately 40% reduction. Beyond this reduction, however, there is a sudden increase in hardness in the two higher-carbon steels, *D*1 and *F*1, whereas curves for the three low-carbon steels, *A*1, *B*2, and *C*1, are regular up to 80% reduction, the highest degree of cold-work reached in these experiments. The low-carbon steels may show a similar increase in hardness if the degree of drawing proceeds still further. Nevertheless, the results confirm the earlier findings and indicate that the later rise in the hardness of cold-drawn wires towards high reductions is influenced by, if not directly concerned with, the carbon content of the steel.

In Fig. 1*a* the five steels were normalized before cold-drawing; in this condition the initial structure would be ferrite and lamellar pearlite. Since carbon was apparently responsible for the anomalous behaviour of the work-hardening properties of the steel, it was thought that the size, shape, and distribution of the cementite particles or lamellae might have some effect upon the hardness curve. To verify this, the following experiments were carried out.

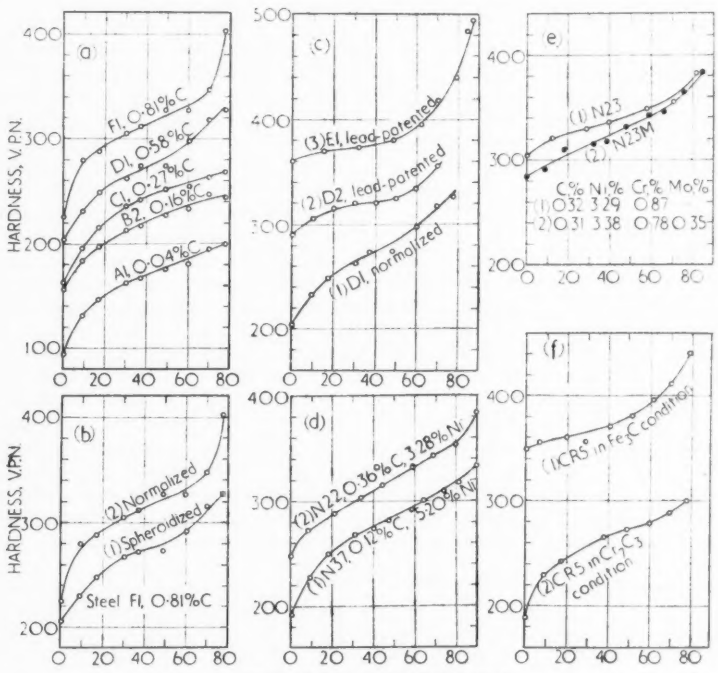
Steel *F*1 was hardened and tempered to give a coarse spheroidized structure in which the carbides were present in large particle form. A number of bars were prepared in this way and the hardness curve obtained on cold-drawn specimens is shown in Fig. 1*b*, curve (1). For comparison, the curve representing the results obtained for the same steel, but initially treated to normalized condition with lamellar pearlite, is replotted as curve (2) in Fig. 1*b*. Although the general shape of these two curves is similar, the hardness corresponding to the same amount of cold-work in the two states is different. With specimens containing spheroidized cementite particles, the sudden increase in hardness occurred earlier than in those initially treated to give a lamellar structure. Moreover, the overall increase in hardness for a given amount of cold-work was different for the two sets of specimens. Although the normalized structure was 20 points harder than that of the spheroidized structure, on cold-drawing to 80% reduction the total increase in hardness for the former was greater, being 180 as compared with 130 V.P.N., which was the corresponding increase obtained with the latter. In these experiments the 'blank rods' were all machined to a given size and the drawing operation which followed was identical in all stages.

To ascertain the effect of coarse or fine cementite lamellae upon work-hardening, experiments were made with steels *D*2 and *E*1. Steel *D*2 was quenched from 850° C. into an isothermal bath controlled at 480° C. It was not possible to suppress the transformation to this temperature, however, owing

**Table I**

**ANALYSES OF STEELS USED IN THE EXPERIMENTS**

| Steel | C,% | Si,% | Mn,% | P,% | S,% | Ni,% | Cr,% | Mo,% |
|---|---|---|---|---|---|---|---|---|
| *A*1 | 0·04 | 0·01 | 0·05 | 0·005 | 0·015 | ... | ... | ... |
| *A*2 | 0·06 | 0·31 | 0·35 | 0·011 | 0·010 | ... | ... | ... |
| *A*3 | 0·07 | 0·01 | 0·04 | 0·009 | 0·054 | ... | ... | ... |
| *B*1 | 0·15 | ... | ... | ... | ... | ... | ... | ... |
| *B*2 | 0·16 | 0·07 | 0·63 | 0·012 | 0·010 | ... | ... | ... |
| *C*1 | 0·27 | 0·21 | 0·60 | ... | ... | ... | ... | ... |
| *C*2 | 0·31 | 0·30 | 0·38 | 0·012 | 0·010 | ... | ... | ... |
| *D*1 | 0·58 | 0·10 | 0·33 | ... | ... | ... | ... | ... |
| *D*2 | 0·59 | 0·28 | 0·45 | 0·010 | 0·011 | ... | ... | ... |
| *E*1 | 0·70 | 0·15 | 0·66 | ... | ... | ... | ... | ... |
| *E*2 | 0·71 | 0·27 | 0·58 | 0·012 | 0·011 | ... | ... | ... |
| *E*3 | 0·77 | 0·13 | 0·62 | ... | ... | ... | ... | ... |
| *F*1 | 0·81 | 0·10 | 0·34 | ... | ... | ... | ... | ... |
| *N*37 | 0·12 | 0·30 | 0·55 | 0·011 | 0·013 | 5·20 | ... | ... |
| *N*22 | 0·36 | 0·31 | 0·56 | 0·013 | 0·013 | 3·28 | 0·16 | ... |
| *N*23 | 0·32 | 0·28 | 0·65 | 0·011 | 0·010 | 3·29 | 0·87 | ... |
| *N*23*M* | 0·31 | 0·25 | 0·51 | 0·010 | 0·017 | 3·38 | 0·78 | 0·35 |
| *CR*5 | 0·20 | 0·17 | 0·25 | 0·011 | 0·012 | 0·06 | 3·02 | ... |
| *CR*4 | 0·51 | 0·20 | 0·38 | 0·014 | 0·013 | 0·06 | 2·96 | ... |




(*a*) Plain carbon steels
(*b*) Effect of initial treatment
(*c*) Effect of initial treatment
(*d*) Annealed nickel steels
(*e*) Oil-hardened and fully tempered alloy steels
(*f*) Steel *CR*5, in $Fe_3C$ or $Cr_7C_3$ condition

**Fig. 1—Hardness curves on cold-drawn specimens**

largely to the rapid nature of transformation of this steel at a higher temperature and to the relatively large sizes of the bars ($\frac{1}{2}$ in. dia.). The structure consisted of rather fine pearlite and some free ferrite. With steel *E*1, rods of about $\frac{1}{4}$ in. dia. were used and the temperature of the bath was slightly lower. The pearlite lamellae obtained were so fine that they were barely resolvable under the microscope. Partly owing to the higher carbon content of *E*1, little free ferrite was present in the specimens. After the treatment, both *D*2 and *E*1 were cold-drawn in the usual way and the hardness results are given in Fig. 1*c*. The curve for *D*1, obtained on specimens with an initial structure of coarse pearlite by normalizing, is replotted to facilitate comparison.

Figure 1*c* shows that, with respect to the work-hardening characteristics of the steel at high reductions, say, beyond 50%, there is little difference between curves (1) and (2). With curve (3), however, the increase in hardness at this stage appears to be more marked than the other two, but this may be due mainly to the higher carbon content of *E*1, rather than to the fineness of the structure in general. On the other hand, the three curves in Fig. 1*c* are different in the early stages of cold-working. Thus, the increase in hardness below 50% reduction was less for curve (2) (*D*2 patented) than for curve (1) (*D*1 normalized), and with curve (3) (*E*1 patented) there was practically no increase up to this point. These results suggest that the work-hardening properties of steel at the early stages are largely dependent on the initial hardness of the specimen. They may be chiefly governed by the conditions of the ferrite and, to some extent, by the state of division and distribution of the cementite. Cold-working hardens the ferrite and breaks down particles or lamellae of cementite. If the ferrite was initially hardened by treatment with the cementite in a finely divided state, as in *E*1, it seems reasonable to expect that further hardening of such a specimen by cold-working at early stages will not be pronounced.

*Low-Alloy Steels*—Figure 1*d*, curve (1), shows the results for a case-hardening, 5% nickel steel, *N*37. Although the carbon content was relatively low, 0·12%, this curve indicates that there was a tendency for a more rapid increase in hardness when the steel was cold-drawn beyond 50% reduction. Comparing this with the plain carbon steels (Fig. 1*a*), it appears that nickel enhances the effect of carbon during cold-working. On the other hand, curve (2), a 3% Ni, 0·36% C steel, *N*22, is parallel with that of *N*37. Although the carbon of *N*22 was higher than that of *N*37, the nickel was considerably lower. It is thus not possible to state whether the effect of increase in carbon content in nickel steels would be of the same order as in plain carbon steels. The initial treatment of *N*22 and *N*37 was annealing at 850° C. and furnace-cooling.

There was no significant difference in the shape of hardness curves for nickel–chromium and Ni–Cr–Mo steels on cold-drawing, as shown in Fig. 1*e*, in which curves (1) and (2) represent the results for *N*23 and *N*23*M*, respectively. The chromium content of these two steels was low, and in both cases the marked increase in hardness beyond about 50% reduction was more marked than during earlier stages of cold-drawing. The initial treatment of both steels was oil-hardening, followed by tempering at 650° C. to obtain a soft structure suitable for cold-working.

The results so far obtained suggest that, in low-alloy steels, the abnormal work-hardening properties are apparent, even with 0·3% C or less. This is not surprising, because alloy elements such as nickel, chromium, or molybdenum, generally lower the eutectoid composition of the steel. Moreover, ferrite containing alloy elements is expected to be harder than that of plain carbon steels. Hence, the effect of cold-work on the increase in hardness would be less at the early stages.

*Effect of Carbide Constitution*—Provided that certain carbide-forming elements are present in sufficient amount to give an adequate alloy/carbon ratio, by suitable treatments the carbon in the steel can be fixed as alloy carbides. In *CR*5 the carbon was present as $Cr_7C_3$ when the specimen was isothermally transformed above 480° C.; below this temperature, similar treatments gave $Fe_3C$.[9] Since the abnormal work-hardening properties were probably due to carbon which was present as cementite in all the steels in the previous experiments, it was thought that a study of the effect of cold-working on *CR*5 after treating to the two different forms of carbide might be most revealing. The carbon content of *CR*5 was relatively low, 0·2%; *CR*4 would have been better (0·51% C) but, in the 'cementite' condition, these specimens were too hard to be cold-worked to any appreciable extent.

Curve (2) in Fig. 1*f* shows the hardness results obtained on *CR*5, which was isothermally treated at 700° C. to $Cr_7C_3$ before cold-drawing, the hardness being about 190 V.P.N. The rate of work-hardening was rapid at the early stages, comparable with that observed with a plain carbon steel drawn from a fully softened state. The rate decreased progressively with increasing reductions up to 60%; after this, the curve indicates only a small rise. The same steel was initially transformed in an isothermal bath at 350° C., when the carbon was mainly $Fe_3C$ and most of the chromium would remain in the ferrite. The general structure was much finer and the initial hardness was higher, about 350 V.P.N. Curve (1), Fig. 1*f*, shows that, on cold-drawing, there was very little increase in the hardness of the specimen up to 40% reduction, but a marked increase beyond this. From the previous results on plain carbon steels, the marked difference between curves (1) and (2) in the early stages might be attributed to the different initial hardness of the specimens concerned, but at high reductions the work-hardening may be influenced by the difference in carbide constitution.

**Wires after Tempering**

*Plain Carbon Steels*—The effect of tempering on the hardness of a cold-drawn low-carbon steel (*A*2) is shown in Fig. 2*a*. Bars of about 1 in. dia. were cold-drawn to $\frac{1}{2}$ in. dia., with a corresponding reduction of 70%. A number of specimens were prepared and subsequently tempered *in vacuo* at different temperatures for 2 hr. The curve in Fig. 2*a* shows that there was only a slight decrease in hardness

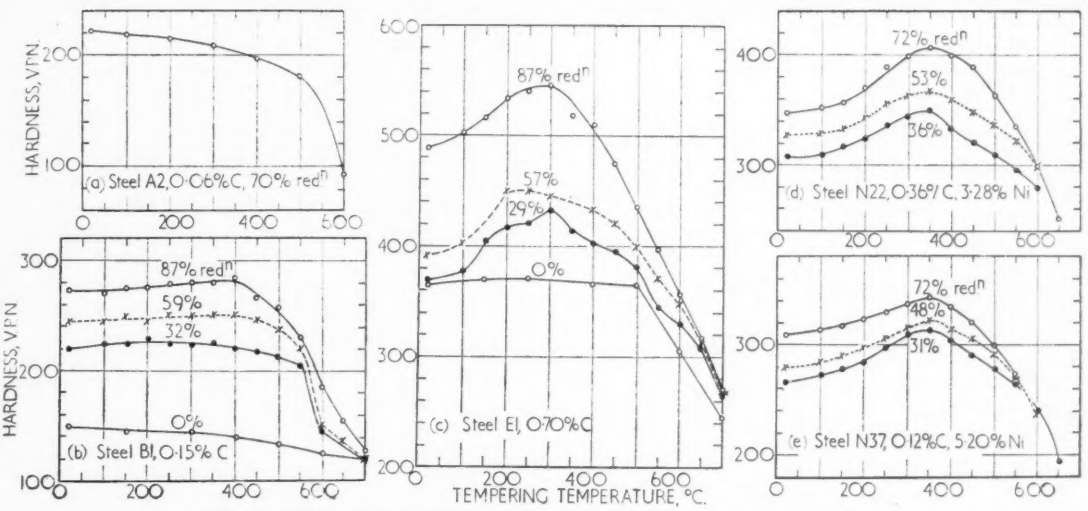



**Fig. 2—Effect of tempering on the hardness of cold-drawn specimens**

on tempering up to 500° C. Above this temperature the decrease was very marked, indicating that recrystallization had become effective. These results are somewhat different from those reported for similar material by other workers. For example, Balicki[10] found a small increase in hardness when Armco iron wires were tempered at moderate temperatures.

The effect of tempering cold-drawn specimens of low-carbon steel *B*1, at different temperatures, is shown in Fig. 2*b*. A normalized specimen, which had not been cold-worked, was also tempered and the results are included for comparison. There was no significant change, apart from a slight decrease in hardness after tempering at successively higher temperatures. With cold-drawn wires, however, a marked drop in hardness invariably occurred above 500° C., and at a lower temperature as the degree of cold-drawing became greater, which is characteristic of the recrystallization phenomenon in cold-worked metals and alloys. Below 450° C. the two wires drawn 32% and 59% reductions respectively, showed no change in hardness, whereas the most severely cold-worked one (87% reduction) increased about 10 points at 400° C.

With the high-carbon steel *E*1 in the patented condition before drawing, the tempering of cold-drawn specimens at low temperatures invariably brought about a marked increase in hardness, the maximum effect being usually at 300° C. Figure 2*c* shows that, after tempering at 300° C., an increment of as much as 60 points was obtained for the three cold-drawn wires, irrespective of their different reductions. The hardness of the undrawn wire was unchanged on tempering up to 500° C., but above this temperature it decreased. That is to be expected because the specimen, although not cold-worked, was initially heat-treated to a semi-hardened condition by patenting. Only when the tempering temperature exceeded the patenting temperature (just below 500° C.), could such specimens be softened.

*Alloy Steels*—The effect of tempering on cold-drawn nickel steels was generally similar to that observed with plain carbon steel. The maximum increase in the hardness of cold-drawn specimens of *N*22 after tempering was 40–60 points (Fig. 2*d*), and with *N*37, 30–50 points (Fig. 2*e*). The optimum temperature at which a maximum increment of tempering-hardness occurred, for both *N*22 and *N*37, was 350° C. —50° C. higher than the corresponding temperature for plain carbon steels. The time of tempering (10 min. to 2 hr.) had no appreciable effect upon this optimum temperature, nor upon the increment of hardness. These remarks apply for all the experiments described in this Section.

The results obtained on the Ni–Cr–Mo steel, *N*23*M*, as shown in Fig. 3*a*, agree in general with those for *N*22 (Fig. 2*d*), except that, for a given amount of cold-work, *N*23*M* showed rather less increase on tempering than did *N*22.

*Effect of Carbide Constitution*—Experiments were carried out to investigate the effect of carbide constitution on the tempering-hardness of cold-drawn specimens. As before, steel *CR*5 was employed and the results are given in Figs. 3*b* and 3*c*. In Fig 3*b*, for which the carbon was present as $Cr_7C_3$ as a result of the initial treatment, the increase in hardness of cold-drawn specimens after tempering was generally small, only 10–15 points even for the most severely worked specimen. With the same steel in the $Fe_3C$ condition, the increase in the tempering-hardness was definitely greater. As shown in Fig. 3*c*, the increment was 20–35 points, depending upon the initial reduction.

The fact that tempering a cold-worked steel at moderate temperatures gives rise to a secondary-hardening phenomenon is not new. Further, the higher the carbon content, the greater was the increase in the strength of the steel after reheating to 300° C. The foregoing results have confirmed that the secondary hardening was mainly associated with the presence of carbon in steel, and have indicated that the condition

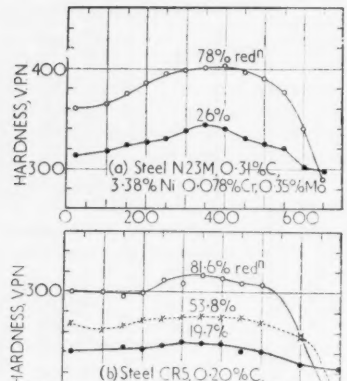



**Fig. 3—Effect of tempering on the hardness of cold-drawn specimens**

of the carbon, for example, cementite or alloy carbides, is important. The secondary hardening may be closely connected with the abnormal increase in hardness of the steel at the later stage of cold-drawing, as shown by the work-hardening curve, since both effects are associated with carbon and are influenced by carbide constitution. They may even arise from the same cause and mechanism. These considerations may seem to be speculative at this stage but will be discussed later in the light of other results.

## DILATATION EXPERIMENTS

### Plain Carbon Steels

In the earlier paper,[8] certain interesting results were obtained on cold-drawn carbon steels by dilatometric measurements. Owing to the large size of the specimens it was not possible to cold-draw much beyond 50% reduction, especially if the carbon content was high. Since, on work-hardening, the abnormal properties were more apparent when the degree of cold-drawing exceeded this stage, it was considered desirable to follow up this investigation with smaller wires to enable cold-drawing to be carried to much higher reductions. In the present work, wires were usually cold-drawn from blank rods of about 0·25 in. dia., with the exception of one or two larger sizes.

Since the dilatometer used in the earlier work was unsuitable for the smaller and varying sizes of wires, a new dilatometer was constructed on the same principle as that employed by Allen, Griffith, and Pfeil,[11] and described in detail in a previous paper.[9] The only modification was that the silica tube, which forms the main part of the dilatometer, was not directly cemented to the dial-gauge attachment, but to a brass adaptor which could be screwed into

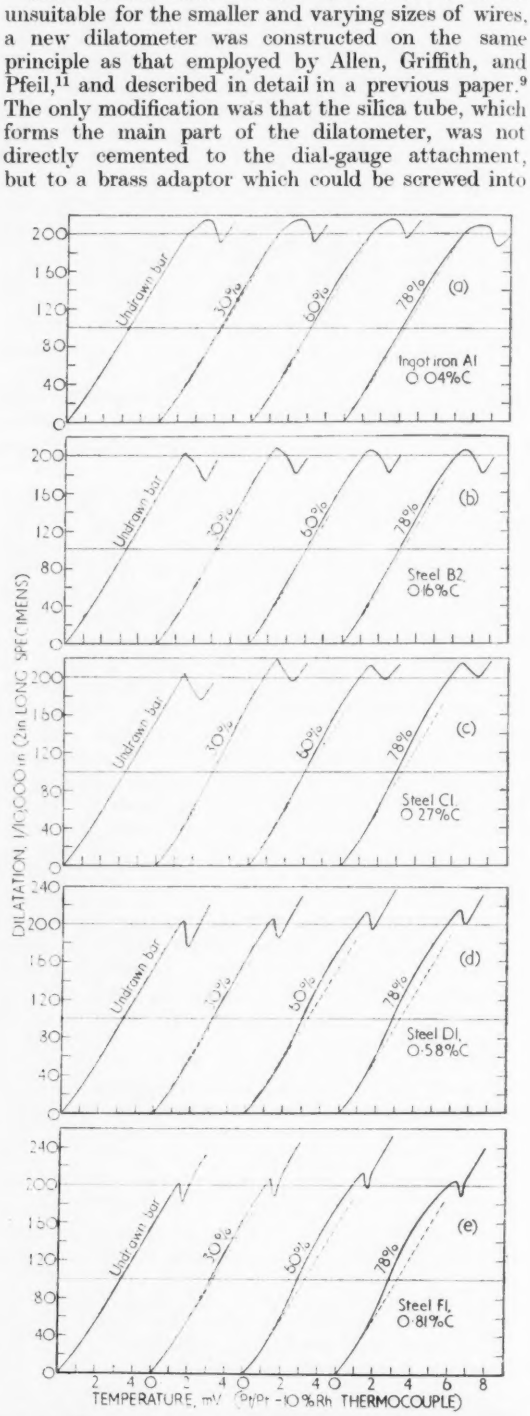



**Fig. 4—Dilatation curves on heating cold-drawn specimens initially in the normalized condition**

the base-plate of the dial-gauge support. This modification was made so that silica tubes of different diameters, chosen to suit wires of different sizes to prevent bending of the wires, were interchangeable with the same dial-gauge attachment. Specimens 2 in. long were used, and the temperature was measured by a Pt/Pt–10% Rh thermocouple inserted through the bottom ring-platform to make contact with the specimen.

Dilatation curves for a series of carbon steels after cold-drawing to various reductions are shown in Fig. 4. Curves for undrawn, normalized specimens are included for comparison. As might be expected, the undrawn specimens showed no change in dilatation and the curves were approximately linear up to the $Ac_1$ temperature. With the cold-drawn wires, however, the following dilatation changes were observed :

(1) An expansion between 150° and 450° C.
(2) A small, gradual contraction between 500° C. and the $Ac_1$ temperature
(3) The contraction corresponding to the normal $\alpha$–$\gamma$ change on heating was apparently less for the cold-drawn than for the undrawn specimens.

These changes are more marked with increasing degree of cold-drawing and still more so with the higher-carbon steels.

The most outstanding feature was the abnormal expansion between 150° and 450° C. on heating. As this expansion took place within the temperature range at which an increase in hardness of the cold-worked steel occurred, it was thought that the nature of this change might throw some light on the cause of secondary hardening. In the earlier paper[8] this was attributed to the decomposition of retained austenite formed during cold-working. Further experiments were carried out to study the dilatation changes in greater detail, along with other properties of cold-drawn steels. For this purpose, steels *E*1 and *B*1, with 0·7% and 0·15% C, respectively, were chosen, largely because they were available in great quantities. Before cold-drawing, *E*1 was patented in an isothermal bath to give a very fine pearlitic structure, but *B*1 was normalized as usual. The dilatation curves obtained on specimens of *E*1 and *B*1, immediately after cold-drawing, are given in Fig. 5.

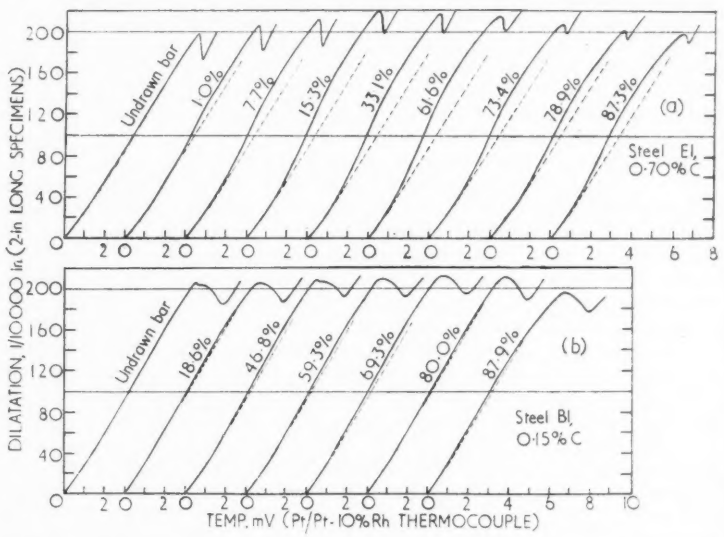
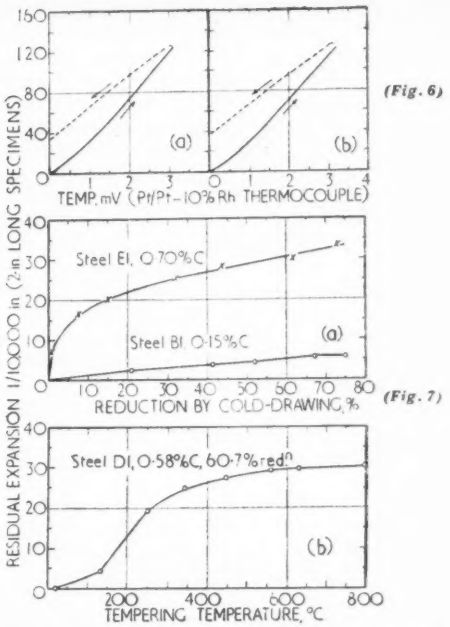



**Fig. 5—Dilatation curves on heating cold-drawn specimens (*a*) initially lead-patented at 480° C. and (*b*) initially in the normalized condition**




(*Fig. 6*)

(*Fig. 7*)

**Fig. 6—Dilatation curves obtained on steel *E*1 (73% reduction) : (*a*) Heated to 450° C. and cooled immediately, and (*b*) heated to 450° C., held for 45 hr., and cooled**

**Fig. 7—Residual expansion of cold-drawn specimens after tempering (*a*) at 450° C. and (*b*) at various temperatures**

Bearing in mind the carbon contents of *E*1 and *B*1, these results are in general agreement with those for the other carbon steels (Fig. 4). Thus, the three dilatation changes listed above are marked, especially in *E*1 (Fig. 5*a*). Moreover, there is, in certain cases, a suggestion of a small contraction between 100° and 150° C., *i.e.*, immediately preceding the abnormal expansion.

To deal first with the abnormal expansion between 150° and 450° C., cold-drawn specimens 2 in. long were heated to, and subsequently cooled from, 450° C., during which the dilatation change was followed. Typical heating and cooling curves obtained on these wires are shown in Fig. 6, in which (*a*) a cold-drawn wire of *E*1 of 73% reduction was heated to 450° C.

and immediately cooled, and (*b*) a similar specimen was heated to the same temperature, but held at the temperature for 45 hr. and then cooled. There was a permanent increase in the length of the wire after the treatment. In both cases the cooling curve was linear, showing that no change was taking place during cooling. Moreover, the fact that the permanent increase in the length of the specimen after heating to, and cooling from, 450° C. was approximately the same in both cases, in spite of the different times of tempering, indicates that the change in the length of the cold-drawn specimen occurrred almost instantaneously during heating and was primarily concerned only with the temperature. If, after the first heating, the same specimen was reheated and the dilatation was recorded, the dilatation curve followed a normal linear course as if the specimen had not been cold-worked until the first heating temperature was reached. Above this temperature the specimen would then resume its cold-worked behaviour.

When the different wires of both *E*1 and *B*1 had been investigated in this manner, it was possible to draw curves for the two steels, showing the relation between the increase in length of the specimen resulting from tempering at 450° C. on the one hand, and the degree of cold-drawing on the other. The curves are shown in Fig. 7*a*, from which it will be seen that, in all cases, the net expansion was much greater for *E*1 than for *B*1. For example, in the cold-drawn wire of 75% reduction, after tempering at 450° C. the increase in length of the specimen was 0·16% for *E*1 and 0·03% for *B*1.

In an attempt to correlate this expansion with tempering temperature, a specimen of *D*1, cold-drawn 60·7% reduction, was employed. Steel *D*1 was used instead of *E*1 mainly because *E*1 was not available in large sizes, so that after severe cold-drawing the diameter would be too small. A reasonably large specimen was used so that, on repeated heating and cooling from different tempering temperatures, there would be no error from bending or creeping. The diameter of the cold-drawn specimen of *D*1 was 0·232 in., and the steel was normalized before drawing. The specimen was placed in a vacuum dilatometer and the total increase in length after cooling from each successive tempering temperature was recorded. The results are given in Fig. 7*b*.

As might be expected, the increase in length of the specimen was rapid between 200° and 300° C. The expansion almost reached its limit at about 450–500° C., and above this temperature very little further increase was observed. The results indicate that the increase in length gained by the cold-drawn specimen on tempering at up to 500° C. was a permanent effect, and the new length was maintained in spite of further heating and cooling, even above the $Ac_1$ temperature. The experiment was repeated and identical results were obtained. If, however, cold-drawn specimens of higher reductions (above 70%) were used, there were indications that the specimen may contract with repeated heating and cooling above 600° C., but this contraction was very small in comparision with the increase in length of the specimen at lower temperatures.

From the increase in the length of cold-drawn high-carbon steel wires resulting from low-temperature tempering, it is clear that such an expansion could not be explained on the basis of the decomposition of retained austenite, as postulated in the earlier paper.[8] If it is assumed that the increase in length must represent a proportional change in the volume of the specimen, a linear expansion of 0·15% would require some 50% retained austenite in the cold-drawn specimen to account for the results. Clearly this was not the case.

The most likely cause of this low-temperature expansion was therefore thought to be mainly a directional change associated with the cold-drawn wires. To verify this, a specimen of *D*1, similar to that employed for Fig. 7*b*, but with a larger diameter (0·325 in.), was carefully measured along its cross-section by a special Mercer gauge, reading directly to 0·001 mm. After tempering at 450° C. *in vacuo*, the diameter of the specimen was again measured and there was a contraction in diameter of about 0·05%. This contraction can be compared with the increase in length of about 0·14% after the same treatment (*see* Fig. 7*b*). They are very nearly of the same order, showing that the volume change of the cold-drawn specimen brought about by tempering up to 450° C., if any, could be only very small. This observation was confirmed by density measurements, as will be described later.

Thus the observed dilatation change between 150° and 450° C. in cold-drawn steel represents a change in the *shape* rather than in the volume of the specimen. Such a phenomenon could only be connected with a redistribution of internal stresses. The internal stresses in a cold-worked steel are of a higher level than in cold-worked iron because the cementite in the steel must cause certain obstruction in plastic flow, and its restraining effect upon the surrounding ferrite matrix will give rise to a greater residual stress in the mass after cold-working. This is borne out by X-ray measurements, as shown in a later Section. On heating a cold-drawn specimen, the redistribution of such residual stress can take place at relatively low temperatures, say, below 450° C. At low temperatures, the steel may be relatively non-plastic and therefore more difficult to yield, but, in view of the difference in the coefficients of expansion of cementite and ferrite, there will be room for readjustment which will release some of the residual stresses.

Assuming that the restraining effect of cementite upon its surrounding ferrite is mainly responsible for the longitudinal expansion of cold-drawn high-carbon steel wire after tempering, then, for a given steel, the shape and alignment of the cementite present—as lamellae or as round particles, for example—might be expected to have some influence upon the dilatation properties of cold-worked specimens. To test this, the following experiments were carried out. Steel *F*1 (0·81% C) was quenched and subsequently fully tempered at 650° C. for a very long time, to give a coarse sorbitic structure in which the cementite was present mostly as large round particles. After this treatment the steel was cold-drawn, and the dilatation curves on specimens of different

reductions are shown in Fig. 8. The difference in dilatation between the cold-drawn and the undrawn specimens was small, particularly for the longitudinal expansion between 150° and 450° C. Bearing in mind the results given in Fig. 4*e*, for the same steel cold-drawn from a lamellar structure, it is clear that the shape of the cementite had a most profound influence upon the dilatometric properties of cold-drawn steel.

It may be relevant to mention that the dilatation change between 150° and 450° C., as shown to be mainly directional, can also be correlated with X-ray results, to be described in detail later in this paper. With a high-carbon, cold-drawn steel, the ferrite was unmistakably in a state of unbalanced internal strain so that its lattice parameter differed in different directions. On heating such specimens, the ferrite lattice expanded along the longitudinal axis of the wire, but contracted in the transverse direction. The temperature of such a recovery and the order of the lattice change observed are in good agreement with the dilatation results already given.

Another dilatation change revealed by Figs. 4 and 5 was the gradual contraction preceding the normal $Ac_1$ transformation. This contraction apparently spread over a wide range of temperature and generally commenced at a lower temperature as the degree of cold-drawing was increased. The lowest temperature, however, was never below 500° C., under the experimental conditions. Moreover, the contraction

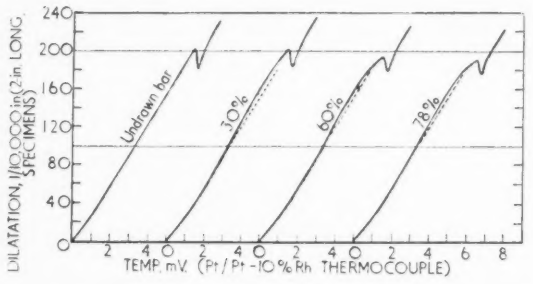



**Fig. 8—Dilatation curves on heating cold-drawn specimens of steel *F*1, 0·81% C. Specimens initially spheroidized by subcritical annealing**

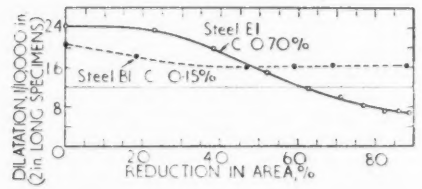



**Fig. 9—Variation of $\alpha$–$\gamma$ contraction for various cold-drawn specimens of steels *B*1 and *E*1**

was more pronounced with increasing degree of cold-drawing and, for a given reduction, this effect seems to be greater for higher-carbon steels.

There is every reason to believe that the gradual contraction occurring between 500° C. and the $Ac_1$ temperature, as already described, is closely associated with recrystallization. Thus, the fact that the gradual contraction commenced at a lower temperature for specimens cold-drawn to a higher reduction is in keeping with the well-known characteristics of the recrystallization phenomenon associated with cold-worked metals and alloys. Since cold-working lowers the density of the steel, which on recrystallization is recovered, when cold-worked specimens are heated to the recrystallization temperatures a contraction is to be expected. As is shown later, the decrease in density on cold-drawing was generally greater for a high-carbon steel (*D*1) than for a low-carbon iron (*A*1). This comparison was based on similar degrees of cold-drawing and was valid up to 62% reduction, beyond which density data for the high-carbon steel were not available. On subsequent tempering, the density of the cold-drawn specimens began to increase when the tempering temperature exceeded 400° C. and was fully recovered at 700° C. in the high-carbon steel. With the low-carbon iron (*A*1), however, this recovery took place much more slowly and the density of the cold-drawn wire remained below that of the undrawn specimen even after annealing at 1000° C. These facts fit in very well with the gradual contraction between 500° C. and the $Ac_1$ change, as given by the dilatation experiments.

Perhaps the least understood feature of dilatation changes is the effect of cold-drawing upon the normal $\alpha$–$\gamma$ contraction. As shown in Figs. 4 and 5, the $\alpha$–$\gamma$ contraction, as given by dilatation in the longitudinal direction, was invariably less for cold-drawn than for undrawn specimens. Moreover, the effect was greater for a high-carbon than for a low-carbon steel. Values of the $\alpha$–$\gamma$ contraction for *E*1 and *B*1, as given in Figs. 5*a* and 5*b*, are plotted in Fig. 9 against the degree of cold-drawing. For 2-in. long specimens, *E*1 and *B*1 gave 0·00245 in. and 0·0021 in., respectively, for the $\alpha$–$\gamma$ contraction in the unworked condition. In both cases this contraction was less after cold-drawing. With *B*1, the $\alpha$–$\gamma$ contraction of the cold-drawn specimens reached its minimum limit at about 50% reduction, being 0·0016 in., compared with 0·0021 in. for the undrawn specimen. With *E*1 the effect was even more astonishing. For the specimen cold-drawn 80% reduction, for example, the observed contraction at the $\alpha$–$\gamma$ change on heating was only 0·0007 in., which is less than one-third of the value for the corresponding unworked specimen. As shown in Figs. 4 and 5, cold-drawing apparently has no effect upon the temperature of the $\alpha$–$\gamma$ transformation of the steel if the beginning and ending of the contraction can be taken as a criterion to indicate the range of this transformation.

In view of the relatively high temperature at which the $\alpha$–$\gamma$ transformation takes place, there seems to be no question of residual lattice strain in the specimen causing the observed difference. On the other hand, preferred orientation of the ferrite in cold-worked specimens is known to exist up to the $Ac_1$ temperature and, on transforming from $\alpha$ to $\gamma$, it might involve the least change of lattice spacing along the longitudinal direction of the wire, the bulk contraction in volume being unaltered. This would mean a greater contraction in proportion

along the diameter of the wire during transformation. Owing to the small size of the hard-drawn wires, it has not yet been possible to test this suggestion with sufficient experimental accuracy, but in any case it does not explain the fact that, on cold-drawing, the $\alpha$–$\gamma$ contraction was much more affected in high-carbon than in low-carbon steels.

Another possibility is graphitization. It is, however, most unlikely that the amount of graphite involved would be sufficient to account for the difference observed.

Even under normal circumstances, the beginning and ending of the $\alpha$–$\gamma$ contraction, as given by dilatation, do not necessarily represent the beginning and ending of the transformation. The question therefore arises whether the temperature of the transformation was in fact affected by cold-drawing. On the assumption that, in cold-drawn steel, the $\alpha$–$\gamma$ transformation takes place earlier—in other words, it commences before reaching the normal $Ac_1$ temperature—it may well be said that the observed effect of the $Ac_1$ point and the gradual contraction preceding it agree. Against this is the fact that when cold-drawn steels were heated to, and subsequently quenched from, just below the normal $Ac_1$ temperature, no hardening effect was observed. In addition, the temperature–resistivity curves for cold-drawn specimens during heating, as described in a later Section, also failed to give any indication to support such an assumption.

The next point to be considered is the fibre or banded structure which persists even after recrystallization. On continuous heating, as in the dilatation experiments, there is little doubt that the banded structure will remain up to the normal $Ac_1$ temperature. At this temperature, the pearlite transforms, but the banded ferrite, being the last to disappear, may form a 'bridge' to offset the contraction. This would mean that the contraction associated with the $\alpha$–$\gamma$ transformation is spread out towards the end of the change when ferrite finally disappears. The modification of the $\alpha$–$\gamma$ contraction in this way does not imply an alteration in the transformation rate, nor any difference in the range of transformation temperatures. Such an explanation appears to be more in accordance with other results discussed later.

The dilatation results so far described agree with those reported in the earlier paper,[8] although two points may be noted. Firstly, in the earlier work, there was no mention of any effect of cold-drawing upon the $\alpha$–$\gamma$ contraction, and secondly, the small contraction between 100° and 150° C., which was observed in cold-worked, low-carbon steels in the earlier work, has not been touched upon here. In the earlier work, cold-drawing was possible only up to 50% reduction. With such relatively low reductions the effect of cold-drawing upon the $\alpha$–$\gamma$ contraction, even for high-carbon steels, was small, and therefore had been overlooked. If, however, the dilatation curves of the earlier paper are carefully examined, it will be seen that such an effect was nevertheless apparent.

With regard to the second point, it may be recalled from the previous paper[8] that the small contraction at 100–150° C. disappeared on ageing. On the other hand, in Fig. 5, in which the curves were taken more or less immediately after cold-drawing, there is only a faint suggestion of such a contraction occurring during heating, even for the most severely cold-worked specimen of the low-carbon steel, *B*1. In view of the results now available, the authors are inclined to think that the small contraction between 100° and 150° C. which was observed only in freshly cold-drawn specimens, as shown in the earlier work, may be mainly associated with the recovery of the elastic strain. The fact that this contraction was not apparent in the present work might be attributed to the different size of specimens and the different method by which the dilatation was transmitted. In the earlier work, large specimens were used and it was possible to drill a $\frac{3}{16}$-in. dia. axial hole halfway down the specimen for the insertion of a thermocouple. The dilatation was transmitted through a hollow silica tube resting on the 'rim,' and the properties measured would be more representative of the surface portion rather than the bulk of the specimen. As shown in a later Section, the stress distribution in a cold-drawn specimen of a low-carbon iron varied from surface to centre across the section and in the longitudinal direction, tensile strain being the greatest near the surface. In a freshly drawn specimen, the elastic strain may be similarly related. If this is so, it will be quite understandable that the dilatation effect will be most marked when such measurements are confined to the surface portion of the specimen.

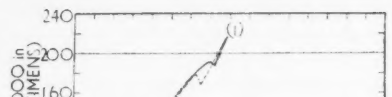



**Fig. 10—Dilatation curves obtained on cold-drawn specimens of steel *E*3, 0·77% C, 97·8% reduction**

Whilst the dilatation change at 100–150° C. (which was usually small and disappeared on ageing) may be associated with the recovery of the elastic strain in the cold-drawn specimen, in certain cases this contraction was very pronounced and apparently did not vanish with time at room temperature. An example of this was a wire of a 0·77% C steel (*E*3), cold-drawn 97·8% reduction in area. This wire was supplied by the National Physical Laboratory with a series of specimens of the same steel cold-drawn to lower reductions. All of them had been aged for an unknown period, probably several months, before the dilatation experiments could be carried out. The dilatometric properties of this set of wires, with the exception of the most severely cold-drawn wire (97·8% reduction), were similar to those of *E*1, Fig. 5*a*. With the last wire, however, a very marked contraction occurred on heating around

150° C., as shown by curve (1), Fig. 10, otherwise the curve was not very different from the others. As this wire had been aged for several months and the contraction at 150° C. did not disappear, it is extremely probable that this particular change is not attributable to the elastic strain recovery.

Owing to the pronounced contraction occurring around 150° C., the net increase in the length of the specimen after tempering at 450° C. was quite small (Fig. 10, curve (2)). In fact, when the wire was heated to, and cooled from, 200° C. the length of the specimen decreased by about 0·05% (Fig. 10, curve (3)). When the same wire was reheated to 450° C. and subsequently cooled, the net expansion was apparently greater (Fig. 10, curve (4)). The difference in the net expansion in curves (2) and (4) corresponded almost exactly with the amount of contraction shown in curve (3). These experiments illustrate the reproducibility of the dilatation results and dispel any suspicion that the marked contraction at about 150° C. in this wire might be a spurious effect due possibly to a slight bending of the specimen owing to its small size (0·06 in. dia. approximately).

The nature of the contraction at 100–150° C. in this case might be similar to the martensitic change associated with quenched high-carbon steels. In view of the drastic cold-drawing given to the specimen and of the high-carbon content of the steel, the intense heat generated by surface friction could lead to the formation of austenite and its subsequent decomposition into martensite. Unfortunately, insufficient wire of *E*3 was supplied to enable further experiments to be carried out to determine whether or not martensite was present in the specimen. However, apart from Trent's finding,[12] that mining haulage ropes contain martensitic surface layers, experiments described later in this paper indicate further that a localized white band was produced in *E*1, as well as in a nickel–chromium steel, through rapid deformation by means of a sudden impact. This band was examined by X-ray and showed the presence of austenite.

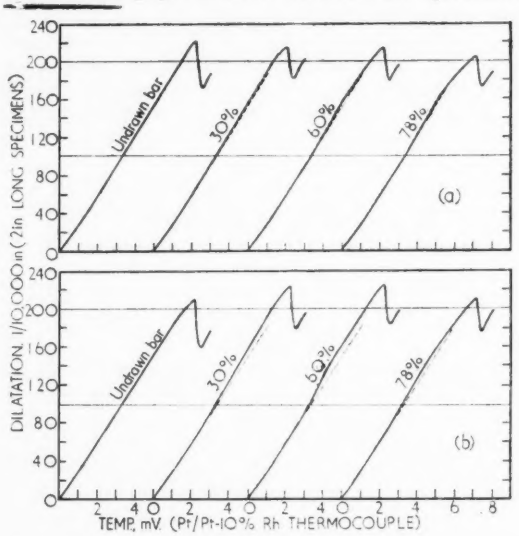



**Fig. 11—Dilatation curves obtained on heating cold-drawn specimens of steel *CR*5: (*a*) in $Cr_7C_3$ condition (isothermally transformed at 700° C.) and (*b*) in $Fe_3C$ condition (isothermally transformed at 350° C.)**

It is hardly necessary to emphasize that the examples cited above are extreme, if not unique, cases. Under sudden impact, the deformation was extremely localized and occurred virtually under adiabatic conditions. With very thin high-carbon steel wires, over-drawing would mean repeated rubbing of a hard surface and might lead to very intense heat as a result of surface friction. The authors do not wish to say at this stage whether the same argument would apply in normal cold-drawing. It is clear, however, in view of the magnetic determinations and other evidence to be given later, that even if austenite is formed during cold-working, the amount retained under normal conditions must be extremely small. In concluding the dilatation results on plain carbon steels, two of the authors would like to stress that, whilst the formation of austenite during cold-working is not entirely without foundation, some of their results in the earlier paper[8] were misinterpreted.

### Low-Alloy Steels

Experiments were also carried out on certain low-alloy steels to study dilatometric properties in cold-drawn specimens. The results may be summarized as follows : With nickel steels, such as *N*22 and *N*37, the dilatation difference between cold-drawn and undrawn specimens was similar to that described for plain carbon steels, commensurate with the corresponding carbon contents. With the Ni–Cr and Ni–Cr–Mo steels, *N*23 and *N*23*M*, respectively, for example, the low-temperature expansion associated with cold-drawn specimens on heating appeared to extend to a slightly higher temperature, *viz.*, 500° instead of 450° C. Other features were similar to those observed with nickel and plain carbon steels.

### Effect of Carbide Constitution

Attempts were made with steel *CR*5 to investigate the effect of carbide constitution upon the dilatometric properties of steel after cold-drawing. Adopting the same procedure as for the hardness experiments, the steel was isothermally treated either at 350° C. to give $Fe_3C$, or at 700° C. to give $Cr_7C_3$, before cold-drawing. The dilatation curves on cold-drawn specimens for the two carbide conditions are shown in Fig. 11. In the $Cr_7C_3$ condition, the difference in dilatation between the cold-drawn and the undrawn specimens was very small, especially as regards the low-temperature expansion (Fig. 11*a*), but in the $Fe_3C$ condition the effect of cold-drawing on the dilatometric properties of the steel was definitely greater (Fig. 11*b*). In the latter case the dilatation changes were comparable with what would have been obtained with a plain carbon steel of an equivalent carbon content.

The treatment temperatures of *CR*5 for the two forms of carbide were not the same, and the shape, size, and distribution of the carbides differed accordingly. With plain carbon steels, the shape and distribution of the cementite have been shown to be particu-

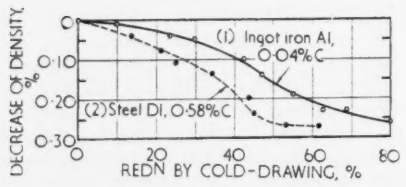



**Fig. 12—Percentage decrease of density on cold-drawing ingot iron *A*1 and steel *D*1**

larly important in determining the dilatometric properties of the steel after cold-drawing, and in view of this, the effect of carbide constitution, as determined from the dilatation results of *CR*5 (Fig. 11), may be somewhat uncertain. Nevertheless, such a possibility should not be overlooked, especially when other properties given later in the paper are taken into consideration.

## DENSITY MEASUREMENTS

Although certain data on the change of density on cold-working are available for iron and carbon steels, experiments were carried out in this direction because it is known that the effect may vary with the material and the mode of deformation. Moreover, the dilatation results required the corroboration of density measurements.

The method of determining density is based essentially upon the difference in the weight of the specimen in two different media, for example, air and a liquid of known specific gravity. Paraffin was used by several previous workers, but in the present experiments distilled water was preferred, mainly because of its greater specific gravity and, what appears to be most important, its smaller coefficient of expansion ; its only disadvantage is the possibility of attack on the specimen, especially if immersed for a long period. In the present experiments long cylindrical specimens weighing 10–18 g. were used ; thermal equilibrium between the specimen and the liquid was reached in a few minutes. Allowing for weighing, the total time of immersion never exceeded 10 min., and there were no signs of any attack.

The specimens were carefully polished, degreased, and cleaned, before suspending in the liquid. Fine silk thread and fine platinum wire were tried for suspension, as by previous workers, but human hair was found more satisfactory. The temperature of the liquid was measured with a thermometer reading directly to 0·1° C., and the weighing was accurate to 0·0001 g. It can be shown by calculations based on the method of least square, that the maximum experimental error was about 0·01%. With paraffin a much more accurate control and reading of the temperature, say to within 0·04° C., would be necessary to obtain the same degree of accuracy.

### Change of Density on Cold-Drawing

Steels *A*1 (0·04% C) and *D*1 (0·58% C) were investigated. After annealing, *A*1 was cold-drawn from 0·41 in. and *D*1 from 0·37 in. dia. The densities of the two annealed steels were 7·864 for *A*1 and 7·839 for *D*1. To facilitate comparisons in the changes of density with respect to the degree of cold-drawing, the results are plotted in terms of percentage decrease, Fig. 12. With *A*1, curve (1), the decrease in density was relatively small below 30% reduction, but beyond this the decrease became more rapid until about 65% reduction when the curve flattens out as if it tends to approach a certain limiting value. With *D*1, curve (2), however, the decrease in density brought about by drawing was marked right from the initial stage and became more rapid with increasing reduction up to only 45%, which corresponds to the inflexion point of the curve. Beyond 50% reduction, further cold-drawing appears to produce no further decrease in the density. These results indicate that, for a given reduction, the decrease in density brought about by cold-drawing was generally greater for the high-carbon steel *D*1, than for the ingot iron *A*1, the maximum difference being at about 45% reduction. On the other hand, from the point of view of maximum decrease in density, irrespective of the degree of cold-drawing, there appears to be little difference between them. Thus, for *D*1 the maximum decrease in density, 0·27%, was reached at about 55% reduction, and for *A*1 the decrease was 0·26% at 80% reduction. With further drawing of *A*1, say to 90% reduction, it appears from the trend of the curve that a decrease of 0·27% in density could be reached, if not exceeded.

### Recovery of Density of Cold-Drawn Specimens on Tempering

Two cold-drawn specimens, *D*1 (60·7% reduction) and *A*1 (62·5% reduction), were chosen for the study of the effect of tempering upon the recovery of density. They were heated *in vacuo* to successive temperatures, held for ½ hr., and subsequently cooled. Density determinations were carried out after each cooling, and the results are shown in Fig. 13 for *D*1 and in Fig. 14 for *A*1.

It should be made clear that the cold-drawn specimen of *D*1 (60·7% reduction) did not belong to the batches used for curve (2) of Fig. 12, but came from the wire used in the dilatation experiments given in Fig. 7*b*. This was to enable the density results to be directly correlated with the dilatation experiments. It will be remembered that this specimen

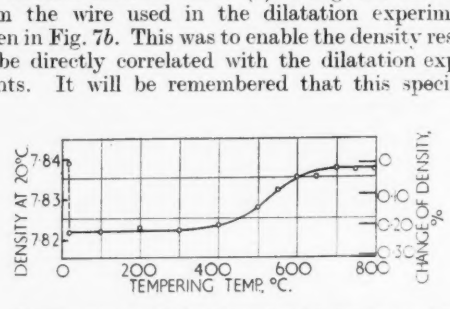



**Fig. 13—Recovery of density of cold-drawn specimen (60·7% reduction) of steel *D*1 after tempering**

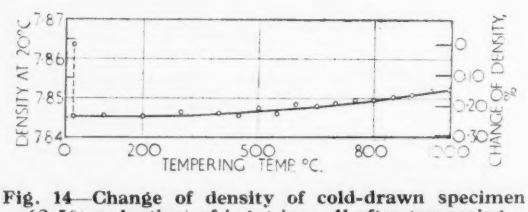



**Fig. 14—Change of density of cold-drawn specimen (62·5% reduction) of ingot iron *A*1 after tempering**

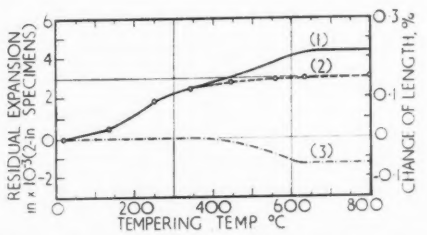
was normalized instead of annealed before cold-drawing. The size of bar and the stages of drawing were also different from those employed for Fig. 12, curve (2). This must account for the fact that the density of the cold-drawn specimen (60·7% reduction), Fig. 13, was 7·822, which is higher than the corresponding value (7·818), calculated from Fig. 12, curve (2).

It will be noted from Fig. 13 that there was no change in density when the cold-drawn specimen of *D*1 (60·7% reduction) was tempered below 400° C. Above this temperature, the density increased and reached a constant value at 700° C. This was very nearly the same as the density of the undrawn specimen, indicating that, as far as the density is concerned, the effect of cold-work upon the high-carbon steel *D*1 was completely removed after annealing at 700° C.

In discussing the dilatation results, the observed expansion when cold-drawn high-carbon steel wires are tempered at 150–450° C. has been attributed to a change in the shape, rather than the volume, of the specimen ; this conclusion is justified by the density results. Further, since the dilatation curve shown in Fig. 7*b* is constant above 500° C., whereas, according to the density results, the specimen should contract, it seems clear that the two processes, the longitudinal expansion due to release of internal stresses and the bulk contraction as a result of the recovery of density, must overlap and therefore compensate each other. Bearing this in mind, the density recovery, as given in Fig. 13, has been resolved into linear contraction and this contraction has been added to the dilatation (Fig. 7*b*) to give Fig. 15.

The full-line curve in Fig. 15 represents the *actual* longitudinal expansion of the cold-drawn specimen of *D*1 (60·7% reduction). In other words, if there were no change in density, the permanent increase in the length of the specimen after heating to different temperatures would be expected to follow this curve. The *actual* longitudinal expansion of the cold-drawn specimen did not finish until about 600° C. This observation is further supported by the X-ray results in a later Section, showing that the unbalanced strains in the ferrite lattice of a cold-drawn high-carbon steel specimen were not completely removed if the tempering temperature was below 600° C.

The density of the cold-drawn specimen of *A*1




(1) Due to directional stress (3) Due to density change
(2) Resultant of (1) and (3) (experimental curve)

**Fig. 15—'Actual' longitudinal expansion (curve 1) of cold-drawn specimen (60·7% reduction) of steel *D*1, as deduced from the observed expansion (curve 2) and the change in density (curve 3) after tempering**

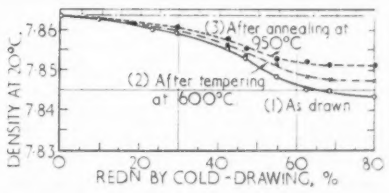



**Fig. 16—Change of density of cold-drawn specimens of ingot iron *A*1**

(62·5% reduction), Fig. 14, was also unchanged when the specimen was tempered below 400° C. Above 400° C. the increase began but proceeded extremely slowly. Thus, the density of this cold-drawn specimen was about 0·23% below that of the undrawn specimen, but even after annealing at 1000° C. for ½ hr. the recovery was only 0·09%. In other words, the density of the cold-drawn and annealed (1000° C.) specimen was 0·14% below that of the undrawn one. This was surprising, especially in view of the results obtained on *D*1, which was completely recovered after annealing at 700° (Fig. 13)—it might be expected that the recovery of the density of the cold-drawn specimen of *A*1 would be accomplished at an even lower temperature. However, the results are in general agreement with those of Ishigaki,[13] who showed that with cold-hammered ingot iron, annealing at 1000° C. recovered only about three-quarters of its lost density, and no explanation was given.

It was first thought that cracks, known to be associated with overdrawn specimens, might be present in this particular specimen of *A*1, and cause the observed discrepancy. Such cracks could not be expected to be healed by annealing. The other six cold-drawn specimens (reductions 30–80%), Fig. 12, curve (1), were therefore annealed, first at 600° C. and then at 950° C., and density determinations were carried out after each annealing. The results are plotted in Fig. 16, which includes curve (1) of Fig. 12 for comparison.

The density values in Fig. 16, for the specimens annealed at 600° or 950° C., lie on a smooth curve with respect to the degree of drawing. For all specimens, the density after annealing at 600° C. was greater than in the as-drawn condition, and further increase was observed on subsequent annealing at 950° C. In no case, however, was the density recovered completely to the value of the undrawn specimen. It was very unlikely that overdrawn cracks were present in all the specimens, expecially those of low reductions ; indeed, micro-examination failed to discover any such gross cracks in any of the specimens.

The presence of submicroscopic cracks in ferrite appears to be refuted by the results obtained on *D*1, because a high-carbon steel would normally be more prone to cracking than a low-carbon iron. Even if submicroscopic cracks do occur in ferrite after drawing, the results obtained on *D*1 indicate that they are completely healed after annealing at 700° C., and there is no reason to suppose that the ferrite in a low-carbon iron would behave differently in this respect.

There were, however, considerable inclusions, mostly iron oxides, in *A*1. If cracks or flaws, submicroscopic or visible, were produced in the inclusions on cold-

drawing, they might contribute to a lowering of the bulk density of the specimen. On this assumption the density of a cold-worked specimen cannot be fully recovered until the cracks or flaws in the inclusions are completely healed or eliminated. Moreover, the decrease in the bulk density brought about by cold-working should, to some extent, depend on the inclusions present. Density determinations were therefore carried out on an undrawn and a cold-drawn specimen of 50% reduction of a different ingot iron, *A*3. Both specimens were then annealed *in vacuo* at 600° C. for ½ hr., and at 950° C. for ½ hr., being tested for density after each annealing. The results were as follows :

| Specimen | Initial Density | Density after Annealing at : 600° C. | 950° C. |
|---|---|---|---|
| Undrawn (annealed) | $7{\cdot}847_4$ | $7{\cdot}847_9$ | $7{\cdot}847_0$ |
| Cold-drawn (50% red.) | $7{\cdot}819_1$ | $7{\cdot}821_2$ | $7{\cdot}821_4$ |

Iron *A*3 contained even more inclusions than *A*1 ; this is supported by the density results, 7·847 as compared with 7·864 for *A*1. For 50% reduction by drawing, the decrease in density for *A*3 was considerably greater, 0·36% as against 0·16% for *A*1, curve (1), Fig. 12. On subsequent annealing, the recovery at 600° C. was only 0·02%, and at 950° C. about 0·04%, out of a total loss of 0·36%. Although the loss of density in this case was very much greater, the recovery in subsequent annealing was comparable with what would have been obtained with *A*1 for a similar reduction of 50% (*see* Fig. 16). These results appear to indicate that, with ingot iron specimens, the decrease in density brought about by cold-working varies with the composition and most likely with the inclusions present.

## THERMO-MAGNETIC PROPERTIES

Magnetic tests can be used for the study of constitutional changes in steel, and are particularly suitable for the detection of austenite or other non-magnetic phases. The most noted methods used recently are the torque balance (Wulff,[14]) and the spring balance (Sucksmith[15]). Although these two methods differ very considerably in detail, the general principle involved in the measurement is the same. Both methods are concerned with the magnetic force acting on the specimen in a non-uniform field with a suitable magnetic gradient. These methods are superior to the orthodox solenoid and ballistic galvanometer arrangement mainly in that the very strong magnetic field used overcomes demagnetization and ensures complete saturation of the material. The last point is very important because, unless the material is brought to complete saturation, or very near to it, magnetic induction is known to be structure-sensitive and the measured quantity cannot be taken to indicate the amount of non-magnetic components in the mass. Wulff,[14] in applying his torque balance method, recommended an ellipsoid specimen for ferro-magnetic material so that the effect of self-demagnetization could be corrected. The applied-magnetic field in Wulff's experiments was only moderately strong, about 2000 gauss, but Sucksmith[15] has used a magnetic field as high as 18,000 gauss to overtake the self-demagnetizing effect of the specimen. With this field an ellipsoid specimen is not necessary. Sucksmith's method is invaluable in the study of ferro- and para-magnetism in metals and alloys where intrinsic properties such as saturation intensity and Curie point are concerned.

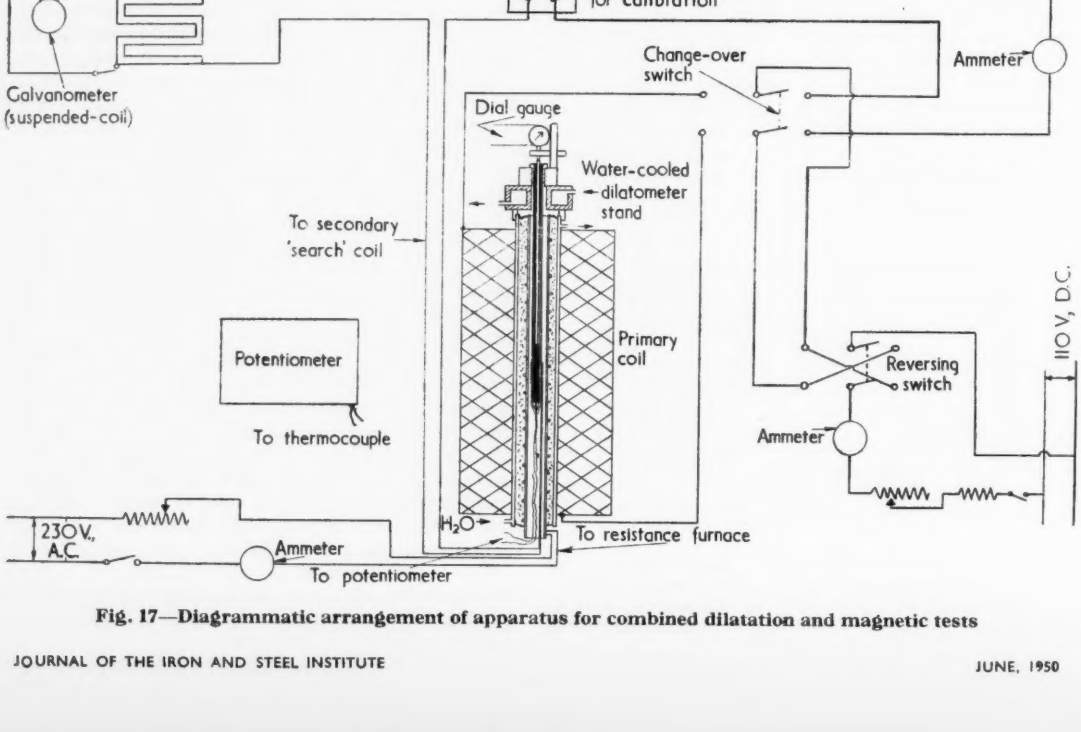



**Fig. 17—Diagrammatic arrangement of apparatus for combined dilatation and magnetic tests**

A disadvantage is perhaps that the specimen is restricted to less than 50 mg. by weight for ferromagnetic material. If the mass concerned is large and the material is heterogeneous, such a small sample may not be representative of the mass.

In view of the heterogeneous nature of steel and the possibility of a localized effect on cold-working, it was considered that cold-drawn wires should be tested over a considerable length. In such a case, the orthodox ballistic method has its advantages. A long specimen and a long solenoid could not be expected to fulfil conditions favourable for complete magnetic saturation, but recent American workers[16] have shown that useful results can be obtained by such a method when a moderate field strength, not less than 500 gauss, is used. With this in mind, it was decided to use the ballistic method, and if interesting results were obtained, the modern method by Sucksmith could be used to check the findings.

The solenoid was 50 cm. long and was wound with 15 layers of 12-S.W.G., D.C.C., copper wire to give 2400 turns. When the coil was excited with a current of 20 amp., the corresponding nominal magnetic field was about 1200 gauss. The former for the coil was a double-walled, brass cylindrical chamber, made of two concentric brass tubes brazed together at both ends. The inner diameter of the former was 2 in. and the external $2\frac{1}{2}$ in. ; the tubes were $\frac{1}{8}$ in. thick, thus leaving a hollow chamber of $\frac{1}{4}$ in. thickness for water circulation. Water-cooling was necessary for the protection of the primary winding because, in carrying out thermo-magnetic experiments, a furnace had to be inserted inside the coil for the heating of the specimen.

The layout is shown in Fig. 17. By incorporating a Mercer dial gauge with the silica tube which bore the secondary coil and was fitted with a fused-silica platform and a transmitting rod, dilatation curves could be taken along with thermo-magnetic curves. The secondary coil, carefully positioned along the middle of the specimen, consisted of 40 turns of fine Nichrome, self-insulating wire, of relatively low-temperature coefficient of resistivity. As with the dilatation experiments, tubes of various bores were selected with appropriate secondary coils to suit the size of wire to be tested.

The specimens for the thermo-magnetic experiments were 0·2 in. or less in diameter and 4 in. long. Longer specimens would seem to be better from the self-demagnetizing point of view, but the length was limited by the uniform-temperature zone of the furnace. The furnace was a non-inductive type, the Nichrome winding being wound in a 'double-back' fashion on a silica tube 24 in. long by 1 in. dia. The $\frac{1}{2}$-in. space between the inner wall of the former of the primary coil and the furnace tube was heat-insulated with asbestos packing. The uniform temperature zone was about 5 in. in the middle along the length of the furnace. The temperature of the specimen was measured by an inserted Pt/Pt–10% Rh couple, connected to a Cambridge potentiometer. Temperature, dilatation, and magnetic readings were taken simultaneously at every 10° C. interval.

As shown in Fig. 17, a standard induction box was incorporated in the magnetizing circuit. By changing the position of the change-over switch, calibration could be carried out without disturbing the arrangement. The ballistic galvanometer was specially designed for high sensitivity and the primary coil was excited by a 110-V., D.C. supply from large batteries. For thermo-magnetic experiments, a current of 14 amp. was used, the safety current of the 12-S.W.G. copper wire being 15 amp. With 14 amp., the corresponding magnetic field was about 850 gauss. For magnetization curves at room temperature, however, the duration of the experiment was shorter, and readings were obtained up to 20 amp. which supplied a field strength of about 1200 gauss.

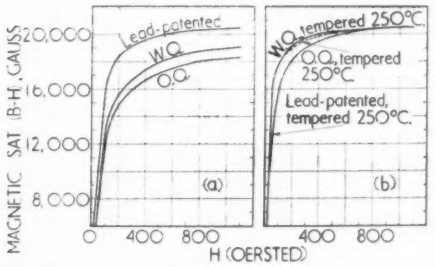



**Fig. 18—Magnetization curves at room temperature for steel *E*1**

Because of the furnace winding and the gap between the secondary coil and the specimen, the readings had to be corrected by subtracting from the experimental results the corresponding 'blank' reading obtained by a 'blank' experiment in which the arrangement was heated and tested at different temperatures without a specimen. For a given furnace winding and secondary coil, the observed 'blank' reading was practically constant irrespective of the temperature; this simplified the calculation for corrections.

Besides the thermo-magnetic experiments, magnetization curves at room temperature were studied. Specimens 6 in. long were used. Since the diameter of the specimens was less than 0·2 in., the length/diameter ratio was at least 30. Corrections for self-demagnetization were carried out by applying the Maxwell equation and using Wurschmidt's data,[17] which are valid for length/diameter ratios less than 100. A separate set of tubes bearing the secondary coil was used, the latter being made of 40-S.W.G. cotton-covered copper wire. The tube was supported along the central axis of the primary coil without the dilatometer attachment and the heating furnace. The gap between the secondary coil and the specimen was measured and the corrections were calculated.

### Magnetization Curves at Room Temperature

As a preliminary experiment, steel *E*1 was investigated in the water-quenched, oil-quenched, and lead-patented conditions. The curves obtained are shown in Fig. 18*a*. The ferritic induction or magnetic saturation of the quenched specimens is less than that of the patented structure. This is undoubtedly due to retained austenite in the quenched specimens. It appears that oil-quenching produced slightly more

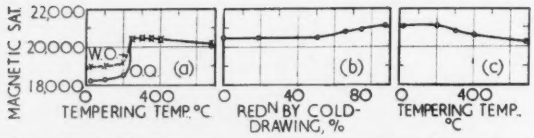



**Fig. 19—Variation of magnetic saturation (Gauss) at 1100 Oersteds (effective field strength) (*a*) on tempering W.Q. and O.Q. specimens of steel *E*1, (*b*) on cold-drawn specimens of steel *E*1, and (*c*) on tempering cold-drawn specimen (87% reduction) of steel *E*1**

retained austenite than water-quenching. The applied magnetic field of only 1100 Oersteds at the maximum, after allowing for self-demagnetization, was not sufficient to saturate the specimen completely. In view of this, the question arises as to what extent the difference in the magnetic saturation obtained could be taken as a direct measure of the amount of retained austenite in the specimen. However, by applying Frölich's law and Kennelly's modification,[18] the magnetization curves could be extrapolated to complete saturation. It can be shown in this way that the degree of saturation at a field strength of 1100 Oersteds was about 95·5% for the quenched and 96% for the lead-patented specimens. With cold-drawn wires, as described later, the corresponding saturation at 1100 Oersteds may vary from 96% for the unworked, to 98% for the worked specimens. If this method of extrapolation is valid, the results of the present experiments should give a reasonably good estimate, say, to within 2 or 3%, of the non-magnetic phases which might be present in the specimens.

The results on quenched specimens after tempering at different temperatures appear to support the above argument. After tempering at a higher temperature, the magnetization curves of the three specimens, lead-patented, water-quenched, and oil-quenched, coincided with one another when the applied magnetic field was greater than 800 Oersteds. This is shown in Fig. 18*b* for the three specimens after tempering at 250° C. Although the curve for the lead-patented specimen differed considerably from those for the quenched specimens at low magnetic field, they became identical at 800 Oersteds or more. The magnetic saturation of the lead-patented specimen did not change to any appreciable extent on tempering, but with the quenched specimens the saturation showed a marked increase between 200° and 250° C. as a result of the decomposition of retained austenite. When the saturation values for the quenched specimens corresponding to a maximum field of 1100 Oersteds are plotted against the tempering temperature, the curves shown in Fig. 19*a* are obtained.

In Fig. 19*a* the increase in magnetic saturation at 200–250° C. was greater for the oil-quenched than for the water-quenched specimen. This is to be expected, since oil-quenching retains more austenite than water-quenching. After 250° C. the saturation values for water-quenched and oil-quenched specimens were identical, but there was a tendency for this to decrease slightly with increase of tempering temperature. For the saturation values corresponding to 300° and 680° C., for example, the difference was about 2½%. This may be due largely to the different degrees of saturation, as shown by extrapolation. Indeed, when the experiments were carried out by the Sucksmith method, where complete saturation was ensured (as will be shown later), there was little difference in magnetic saturation at room temperature between tempered martensite and the fully softened structure.

With cold-drawn wires of *E*1, the magnetic saturation as given by a magnetic field of 1100 Oersteds was unchanged up to 50% reduction by drawing. Beyond 60% reduction, however, it increased slightly with the degree of cold-drawing, as shown in Fig. 19*b*, where saturation values for the different specimens are plotted against the respective reductions. For instance, the specimen cold-drawn 87% reduction apparently showed a saturation value some 3% greater than the undrawn specimen.

On tempering, the saturation value of cold-drawn wires changed very little below 200° C. Above 200° C. it began to decrease very gradually with increasing tempering temperature. A curve relating the observed magnetic saturation of the specimen with tempering temperature on *E*1, cold-drawn 87% reduction, is given in Fig. 19*c*, from which it will be seen that a slight decrease in the observed saturation occurred on tempering above 200° C. Only by tempering at a relatively high temperature, say, 680° C., was the observed magnetic saturation value of the cold-drawn specimen restored to normal, *i.e.*, to the same level as the undrawn specimen. Here again it should be emphasized that the observed difference may be due largely to the different degrees of saturation. Thus, it can be shown by extrapolation that the saturation corresponding to a maximum field of 1100 Oersteds may vary from 96% for the soft- to about 98% for the hard-drawn specimens—a difference of 2%, which is very near the 3% observed as the difference in magnetic saturation between the undrawn and the 87% reduction specimen.

These results indicate that, in approaching magnetic saturation, the behaviour of a hard-drawn wire is rather different from that of a soft specimen, although their true saturation values may be nearly the same. It would appear that the effect of cold-working on steel produced a state of affairs similar to that observed with tempered martensite.

No change in magnetic saturation was found with the low-carbon steel *B*1, either on cold-drawing or after tempering. From this, and the results on the high-carbon steel *E*1, it would appear that the cold-work produces changes in the cementite which may be responsible for the increase in magnetic saturation. Thermo-magnetic experiments were therefore carried out primarily to investigate the cementite change in both quenched and cold-drawn steels.

### Thermo-Magnetic Experiments

The difference in magnetic saturation between cementite and ferrite at room temperature is not large, but the effect can be magnified by raising the temperature and investigating the Curie point of the cementite. Since the Curie point of cementite occurred at about 210° C., whereas that of the ferrite takes place at about 760° C., it is clear that between room

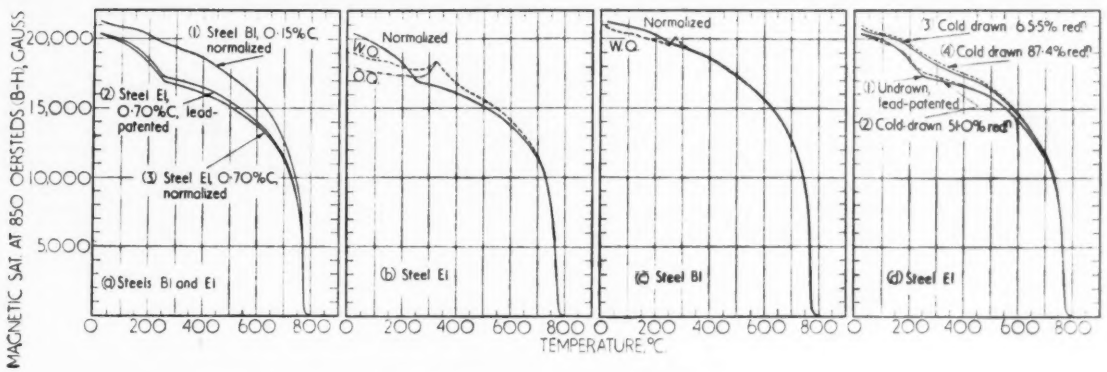



**Fig. 20—Thermo-magnetic curves**

temperature and 210° C. the magnetic properties of cementite are much more sensitive to a change of temperature. For this reason, thermo-magnetic experiments are particularly suitable for the detection of any small changes associated with cementite in cold-worked steels. As already described, a constant magnetic field of 850 Oersteds was used, and the degree of saturation was of the order of 95% for soft specimens, by extrapolation from the magnetization curves obtained at room temperature.

Steels *E*1 and *B*1 were used, and curves are shown in Fig. 20*a* for (1) *B*1 normalized, (2) *E*1 lead-patented, and (3) *E*1 normalized. Because of its considerably lower carbon content, *B*1 gave a higher saturation than *E*1. As might also be expected, the Curie point of cementite was marked with *E*1, but not with *B*1. With *E*1, there was no difference in magnetic saturation for the two specimens at room temperature, but on heating the difference was apparent. Thus, curves (2) and (3) take a slightly different course, although in both cases the discontinuity on the curve corresponding to the Curie point of cementite is sharp and clear-cut. The temperature of the cementite change, as shown in Fig. 20*a*, apparently occurs at about 250° C. This is higher than normal for the $A_0$ point in steel (about 210° C.). It should be pointed out that in the present experiments the thermocouple was in contact with one end of the specimen and not at its centre, and there was a temperature difference between the thermocouple junction and the centre portion of the specimen. For the same reason, the $\alpha$–$\gamma$ change of *E*1 is shown to be at about 770° C. instead of 730° C.

Curves for *E*1, water-quenched and oil-quenched, are shown in Fig. 20*b*, the curve for the normalized specimen being included for comparison. Owing mainly to the presence of retained austenite, the magnetic saturation with the quenched specimens was lower than with the normalized specimen until the temperature was near the Curie point of cementite. Between 275° and 325° C., the sudden increase in magnetic saturation in the two quenched specimens was marked, indicating the decomposition of retained austenite. Here, again, the results show that more retained austenite was present in the oil-quenched than in the water-quenched specimen. After the decomposition of retained austenite at 325° C., the curves for the two quenched specimens became identical, but they are different from that of the normalized specimen in that the magnetic saturation in the corresponding temperature range was higher. The maximum difference was at 325° C., immediately after the decomposition of the retained austenite. As the temperature increases further, the gap between the curve for the quenched specimens and that for the normalized specimen gradually diminishes until just below the $Ac_1$ temperature where they meet, Fig. 20*b*. This is in agreement with the results obtained at room temperature, already described, indicating that, most probably because of incomplete saturation, the observed magnetic induction for tempered martensite was somewhat higher than for a fully softened structure.

Even with the 0·15% C steel, *B*1, water-quenching from 900° C. appeared to give rise to a very small amount of retained austenite. This is shown in Fig. 20*c* by a small increase in magnetic saturation at 250–275° C. Unlike the high-carbon steel *E*1, the curve for the quenched specimen of *B*1 was identical with that of the normalized specimen of the same steel at 300° C. or above. The presence of a small amount of retained austenite in mild steel after quenching was also shown by Wrażej,[19] who used a steel similar to *B*1, oil-quenched from 900° C., and the specimen was examined by X-ray.

The results for cold-drawn specimens of *E*1 are shown in Fig. 20*d*; only a few of the curves are given to avoid overlapping, *viz.*, (1) the undrawn specimen (lead-patented), (2) 51% reduction, (3) 65·5% reduction, and (4) 87·4% reduction. Curve (2) follows closely to (1), showing that, within the range of temperatures tested, the difference in magnetic saturation between the 51% reduction wire and the undrawn specimen was small. This applies to the other wires cold-drawn less than 51% reduction, the results of which are not shown. When the cold-drawing exceeded 60% reduction, however, the specimen showed a slightly higher magnetic saturation than the undrawn specimen—in agreement with the results already described for room temperature. Although this apparent difference may again be attributed mainly to the difference in the satura-

tion attained for the various specimens, it is interesting to note that such a difference increases with temperature and reaches a maximum at 250–275° C. (Fig. 20*d*, *cf.* curves (3) or (4) with (1)). As a result, the cementite Curie point for the severely drawn specimens was not apparent and in this respect the specimens concerned behaved as if the carbon content had been reduced (*see* curves (3) and (4)).

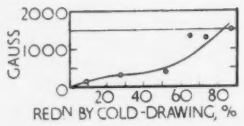



**Fig. 21—Increase in magnetic saturation at the Curie point (250° C.) for cold-drawn specimens of steel *E*1**

Using the curve for the undrawn specimen, Fig. 20*d* curve (1), as a base, the difference in the observed magnetic saturation at the cementite point (250° C.) between the different cold-drawn specimens was compared and is plotted against the degree of cold-drawing in Fig. 21. This curve can be regarded as an enlargement of a previous curve (Fig. 19*b*), relating the variation of the observed magnetic saturation at room temperature with the degree of cold-drawing, In other words, the effect of cold-drawing upon the observed magnetic saturation of *E*1 at room temperature was magnified by taking the corresponding values at the cementite Curie point. This suggests that the observed increase in the magnetic saturation of *E*1 at the later stage of cold-drawing (beyond 60% reduction) may be closely related to a change in the state of the cementite. Bearing in mind the shape of the work-hardening curve of *E*1 (curve (3), Fig. 1*c*), it is an interesting coincidence that the increase in hardness on cold-drawing should be more rapid at the later stage where the magnetic saturation or the cementite Curie point of the steel was noticeably affected.

To investigate whether the cementite Curie point, which disappeared in severely drawn wires, could be recovered by low-temperature tempering, specimens of *E*1 were tested during heating and cooling from about 400° C. Some of the curves are shown in Fig. 22*a*. For the undrawn specimen the cementite point was equally marked on heating and cooling, the hysteresis being very small. With the 65·5% and the 74% reduction wires, however, on heating to, and cooling from, 400° C. the cementite point as revealed by the cooling curves was relatively obscure. There was a suggestion of a very slight recovery because both specimens, on returning to room temperature, showed a slightly lower value in the observed magnetic saturation than before heating. It would appear from Fig. 20*d* that a much higher temperature, say, just below the $Ac_1$ point, may be necessary for the complete recovery of the cementite change in cold-drawn steel.

A comparison of the thermo-magnetic curves from quenched and cold-drawn specimens, Figs. 20*b* and *d*, respectively, reveals one common feature, *i.e.*, above 300° C. both specimens show a higher value in magnetic saturation than the normalized or undrawn specimens. In fact, the curve for the quenched specimen at high temperatures, say, above 350° C., is very similar to the corresponding part of the curve for a hard-drawn wire. Since, with cold-drawn wires, the effect of tempering up to 400° C. on the recovery of the cementite point was small, it was decided to find out whether this would also apply to quenched specimens. Figures 22*b* and *c* represent the water-quenched and the oil-quenched specimens of *E*1. The dilatation curves for the same specimens have been included, from which the decomposition of martensite during heating may be followed. The thermo-magnetic curves obtained on heating to 400° C. are similar to those in Fig. 20*b*, the chief feature being the sudden increase in magnetic saturation between 275° and 325° C., which has been attributed to the decomposition of retained austenite in the quenched specimens. On cooling from 400° C., both thermo-magnetic curves show that the cementite Curie point was not very apparent, although, on reaching room temperature, both specimens had regained their normal value in magnetic saturation. Both dilatation curves obtained on heating showed marked contractions at 120–200° C. and at 300–400° C. The first contraction was undoubtedly due to the decomposition of tetragonal martensite ; no corresponding changes can be seen in the thermo-magnetic curves. The second contraction (300–400° C.),

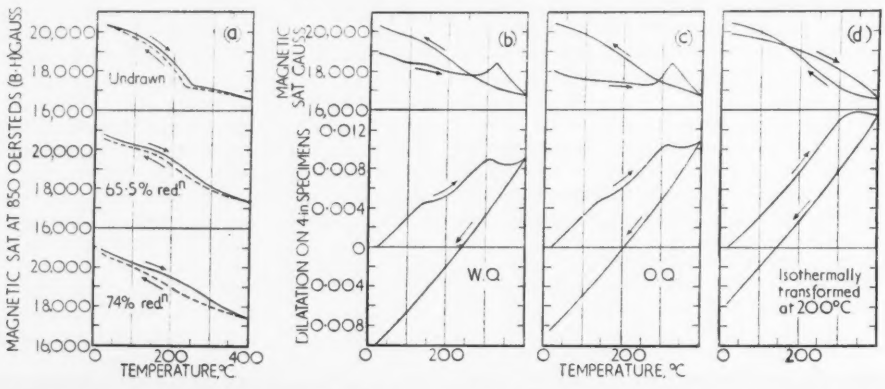



**Fig. 22—Thermo-magnetic and dilatation curves on heating and cooling (400° C.) for specimens of steel *E*1**

recently attributed to a carbide change,[20] is discussed later.

Because of the considerable amount of retained austenite in water- or oil-quenched specimens, the results described above do not indicate whether the cementite point would be entirely absent in pure martensite. The following experiments were therefore carried out. A specimen of *E*1, 4 in. long and 0·2 in. dia., similar to those employed for water- or oil-quenching, was quenched from 850° C. into an isothermal bath at 200° C. After 96 hr. in the bath, the specimen was quenched in brine. Thermo-magnetic and dilatation curves on subsequent heating and cooling are shown in Fig 22*d*. The initial treatment was intended for a completely martensitic structure, but, as will be seen from the thermo-magnetic curves, a trace of retained austenite appears to be present because, after cooling from 400° C., the magnetic saturation was slightly higher. However, the structure was near enough to completely martensitic and the thermo-magnetic curve obtained on heating was perfectly smooth and gave no suggestion whatsoever of any discontinuity corresponding to the cementite change. The cooling curve shows that after tempering at 400° C. there was only a partial recovery of the cementite point, as with the water- or oil-quenched specimens.

Following these experiments, two specimens of *E*1 were sent to the Physics Department for confirmation. One was a cold-drawn wire of about 90% reduction and the other consisted of a martensitic structure obtained by water-quenching from 850° C. and tempering at 250° C. for ½ hr. to decompose the retained austenite. Both specimens were about 1 mm. in dia. and, when broken up into short pieces about 4 mm. long, were suitable for examination by Sucksmith's thermo-magnetic arrangement. The experiments were conducted at a magnetic field of 13,740 Oersteds and complete saturation was ensured. The authors are greatly indebted to Professor W. Sucksmith, F.R.S., for the results obtained on these two specimens, as shown in Fig. 23, where (*a*) is the heating curve for the cold-drawn specimen, (*b*) the curve for the same specimen on cooling from about 800° C., and (*c*) the heating curve for the martensitic specimen. The main features of the results can be summarized as follows :

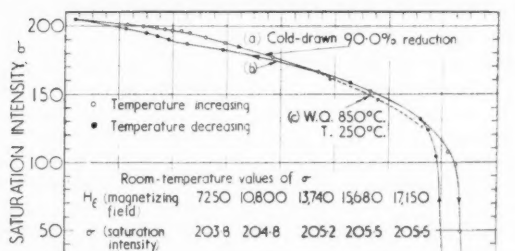



**Fig. 23—Saturation-intensity/temperature curves for cold-drawn (90% reduction) and water-quenched and tempered specimens of steel *E*1. Magnetization field ($H\epsilon$) 13,740 Oersteds**

(1) Magnetic saturation at room temperature is apparently unaffected by cold-drawing, because the cooling curve returns to the point at which the heating curve commences. This also applies to the martensitic specimen. The results appear to preclude the presence of austenite in the cold-drawn specimen, at least not more than ½% if the latter is taken as the limit of experimental accuracy. Bearing in mind the treatment given to the martensitic specimen, the results indicate that tempering at 250° C. decomposed completely any retained austenite in water-quenched carbon steel. On the other hand, whilst the measurement of the magnetic saturation intensity at room temperature alone would give a good indication of the presence or absence of non-magnetic phases such as austenite, it is not sensitive to other constitutional changes, for example, the decomposition of cementite into carbon and free ferrite. Thus, if $\sigma_f = 214$ and $\sigma_c = 130$ are the values of saturation intensities for ferrite and cementite, respectively, at room temperature, then for *E*1, which contains 0·7% of carbon and therefore 10·5% of cementite, the saturation intensity will be :

$$\sigma = 0{\cdot}105 \times 130 - 0{\cdot}895 \times 214 = 205{\cdot}2$$

This is in exact agreement with the room-temperature value in Fig. 23. Assuming that by some unknown processes all the carbon in *E*1 is decomposed into free carbon and ferrite, the amount of ferrite would be 99·3%, and as carbon is regarded as non-magnetic, the new saturation intensity of the steel will be :

$$\sigma' = 0{\cdot}993 \times 214 = 212{\cdot}5$$

The difference between the two values given above is only 3½%. If ½% is taken as the limit of experimental accuracy in the measurement of the saturation intensity, it seems clear that the decomposition of combined carbon in the steel up to as much as 0·1% would not affect the observed value in saturation intensity to any appreciable extent. These remarks, however, apply only to room temperature. At higher temperatures the change of saturation intensity with respect to the decomposition of cementite would be more sensitive, especially above 200° C. when cementite is non-magnetic.

(2) Between room temperature and 500° C. the heating curve (Fig. 23) for the cold-drawn wire (curve (*a*)) coincides completely with that for the martensitic specimen (curve (*c*)). The chief feature is that in both cases the curves are perfectly smooth and show an entire absence of the cementite Curie point. This point is unmistakably marked on the cooling curve (*b*), indicating that the effect of cold-work on the cementite in steel is removed after heating the specimen to above its $Ac_1$ temperature.

(3) Above 500° C. and up to the $Ac_1$ transformation, the thermo-magnetic curve for the martensitic specimen lies below that of the cold-drawn wire during heating. This difference is well outside the experimental error and must be associated with some difference in the constitution of the two specimens. It is well known that work-hardened steel is more prone to graphitization than quench-hardened speci-

mens when heated to sub-critical temperatures. The observed difference might be due to this cause.

The results hitherto obtained indicate that, from the cementite point of view, the effect of cold-drawing on steel seems to be similar to that produced by quenching, followed by low-temperature tempering. In other words, the state of carbon in cold-drawn specimens may be said to be similar to that in tempered martensite. It is generally regarded that in tempered martensite the separation of carbon from solid solution is more or less complete and the precipitated carbides are in a state of very fine dispersion. A similar state may be said to exist in cold-worked steel because cold-work breaks down large carbides into small particles. Whilst such a contention appears to be feasible, the question remains whether fine particle sizes could, in themselves, be responsible for the observed effect. In the authors' opinion, it is difficult to explain the results on the basis of small particle sizes alone, because the Curie point is generally regarded as an intrinsic property which should vary only with the composition or the lattice parameters of the cementite lattice.

It has been suggested[21] that cementite consists of a framework of close-packed iron atoms held together by metallic bonding, the carbon atoms being accommodated in the largest interstices and held in position by bonding which has a certain degree of metallic nature. In other words, cementite, although nominally an intermetallic compound, may have many characteristics of a solid solution. Thus, the composition of cementite may vary with thermal treatment. In line with the characteristics of a solid solution, cementite should be capable of withstanding a certain amount of distortion. This may be particularly true with cementite in steel, where it is embedded and constrained in an iron matrix and any decomposition would be rendered more difficult. We are inclined to think that cementite in cold-worked steel may be in a highly distorted state, but to what extent its properties are different from those of undistorted cementite is very difficult to say. With regard to tempered martensite, it may be interesting to point out that Kurdjumov and Lyssak,[22] in their recent studies of tempered martensite, claimed to have identified a precipitated carbon-rich phase rather different from the normal orthorhombic cementite. Moreover, recent American workers[23] have shown that the precipitated phase from martensite, on tempering, had a hexagonal structure isomorphous with iron nitride. These points are discussed later in the paper.

## MISCELLANEOUS EXPERIMENTS

### X-Ray Examination on Cold-Drawn Wires

The primary object of the X-ray examination was to ascertain whether austenite was present in cold-drawn steels. To detect minute quantities of a second phase, the powder technique would be more accurate than the glancing-angle method, as the phase might be unevenly distributed in the mass.

Very thin cold-drawn wires were suspended in a powder camera and the X-ray was taken in the usual way. The wire was sufficiently thin for the rotation to bring the whole cross-section to contribute towards X-ray diffraction, thus increasing the chance of detection of any second phase.

Steels *B*1, *E*1, *N*22, and *N*23*M* were examined, using wires of about 0·02 in. dia., which had received more than 90% reduction by cold-drawing. Before examination, the wires were etched to remove the surface layer and were carefully straightened to ensure axial rotation. The camera was of the Debye-Scherrer type, 9 cm. in dia., and X-rays were excited with either a chromium or a cobalt target. The results did not show the presence of austenite in any of the cold-drawn wires and, in all cases, only the characteristic ferrite lines were observed.

The absence of cementite lines in the X-ray pattern of the cold-drawn specimen of *E*1 called for further investigation because this steel contained no less than 10% of cementite. The wire was subsequently tempered *in vacuo* at 50° C. intervals for 15 min. and X-ray examinations were carried out after each tempering. Cementite lines were absent up to 400° C., but after tempering at 500° C., they were clearly visible. Heating to a higher temperature, say 600° C. or above, improved the intensity of the cementite lines only very slightly.

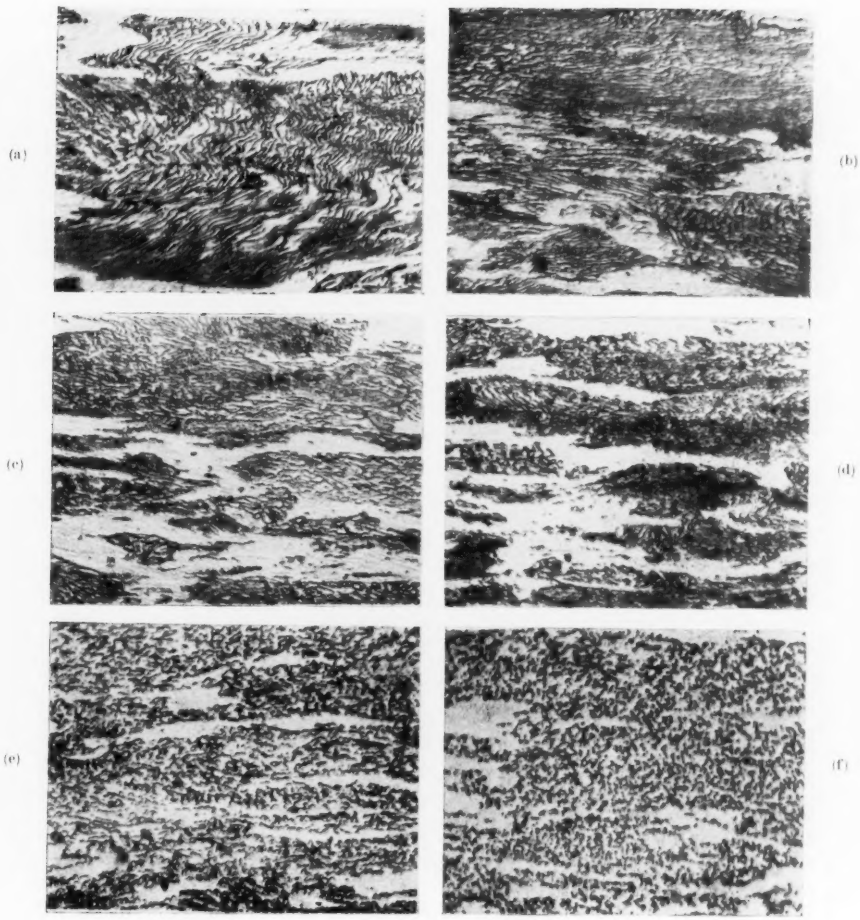
To find at what stages of cold-drawing the cementite lines of *E*1 were most affected and eventually became invisible, a series of cold-drawn wires, about 0·02 in. dia., but corresponding to various reductions by drawing, were examined. The intensity of the cementite lines appeared to be only slightly affected up to 50% reduction; beyond 60% reduction the lines were fainter with the increase in the degree of cold-drawing, until above 90% reduction they were invisible.

The disappearance of the cementite lines in severely drawn wires, as found by X-ray, does not, of course, mean that a considerable amount of the cementite in the steel had been decomposed through cold-working. The major cause of the decrease in intensity, which led to the apparent disappearance of the cementite lines, must be the diminishing particle size and the distortion of the cementite lattice. The scattering due to the distorted ferrite in the matrix may also affect the lines, but this could be only relatively small, because at 50% reduction the distortion of the ferrite must very nearly have reached its usual limit, and the intensity of the cementite lines was apparently reduced only slightly.

### Microstructure

Micro-examinations on cold-drawn wires revealed no constitutional changes, but the elongation of crystal grains in the drawing direction was generally marked. With *E*1 (lead-patented), drawn from an initial diameter of 0·2 in., for example, the elongation of pearlite grains was distinguishable at 30% reduction. This was more marked with increasing degree of drawing, and at 80–90% reduction the wire showed a well-defined fibre structure.

The effect of tempering on the microstructure of cold-drawn steels was also studied. Photomicrographs of *D*1 are shown in Fig. 24. The steel was cold-drawn from a normalized condition, the cementite lamellae being coarser than those of the lead-patented specimen. Even after very severe cold-




(a) As-drawn (70% reduction)

(b) Tempered for ½ hr. at 300° C.

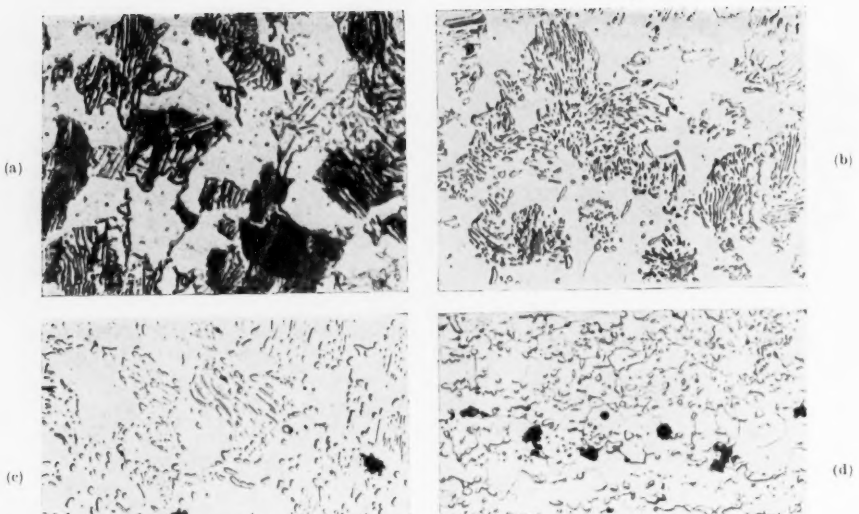
(c) Tempered for ½ hr. at 400° C.

(d) Tempered for ½ hr. at 500° C.

(e) Tempered for ½ hr. at 600° C.

(f) Tempered for ½ hr. at 700° C.

**Fig. 24—Change in microstructure on tempering cold-drawn specimens (70% reduction) of steel *D1* (0·58% C) at various temperatures. Etched in nital × 500**



(a) Fully annealed structure of steel *D*1 in the undrawn condition

(b) As (a), after subcritical annealing at 650° C. for 6½ days

(c) Steel *D*1, cold-drawn 10% and subcritically annealed at 650° C. for 6½ days

(d) As (c), after 10 days at 650° C.

(e) Steel *D*1, cold-drawn 30% and subcritically annealed at 650° C. for 6½ days

(f) Steel *F*1, cold-drawn 16% and subcritically annealed at 650° C. for 6½ days

**Fig. 25—Graphitization in cold-drawn carbon steels *D*1 (0·58%C) and *F*1 (0·81%C), after subcritical annealing in nitrogen atmosphere. Etched in nital × 600**

drawing, it was still possible to resolve the cementite lamellae, although the pearlite grains were distorted and elongated in the direction of drawing. Figure 24*a* illustrates the microstructure of *D*1, cold-drawn 70% reduction, and, within the elongated pearlite grains, some of the cementite lamellae were 'curved' by distortion. On tempering, there was no visible change in microstructure up to 300° C. (Fig. 24*b*), but at 400° C. some of the distorted cementite lamellae tended to break up into smaller segments (Fig. 24*c*). This took place gradually and was increasingly marked with the rise in tempering temperature. Thus, at 500° C., practically all the cementite lamellae in the pearlite grains had been broken up (Fig. 24*d*). At 600° C. the broken cementite particles began to assume a round shape (Fig. 24*e*), but even at 700° C. the round cementite particles appear to remain within the boundaries of the original elongated grains (Fig. 24*f*). The cold-drawn structure was completely destroyed when the specimen was tempered at 800° C. Each specimen was tempered for ½ hr.

Although the microstructures in Fig. 24 do not indicate any constitutional changes, they are interesting from the point of view of orientation of crystal grains; thus cold-drawing gives rise to a fibre structure and tempering above 700° C. destroys it. These changes have a profound effect upon the electrical properties of the steels, as described in a later Section.

**Graphitization in Cold-Drawn Carbon Steels after Subcritical Annealing**

Experiments were carried out to confirm that cold-working promotes graphitization in steel on subcritical annealing. Steels *B*2, *C*1, *D*1, and *F*1 were fully annealed, before cold-drawing, at temperatures appropriate to their carbon contents. Owing to the relatively large size of the bars, 0·4 in. dia., and the fully annealed structure, steel *F*1 could be cold-drawn only to 16% reduction without breaking, and *D*1 to 60% reduction, but a much higher reduction could be obtained with *B*2 and *C*1. After cold-drawing, the specimens were annealed in nitrogen at 650° C. for up to 10 days. In each case, an undrawn specimen was annealed at the same time to serve as control. After this treatment, the cold-drawn specimens of *D*1 and *F*1 contained much graphite, whereas the undrawn specimens were apparently free. Photomicrographs of cold-drawn specimens are shown in Fig. 25. With the low-carbon steels *B*2 and *C*1, no graphite flakes were observed, irrespective of the degree of cold-drawing and the time of annealing at 650° C.

In Fig. 25, (*a*) to (*e*) represent *D*1, and (*f*) represents *F*1. Figure 25*a* gives the fully annealed structure of the undrawn specimen of *D*1 and Fig. 25*b* the structure after subcritical annealing at 650° C. for 6½ days. No graphite flakes are visible. Graphite is present in Fig. 25*c*, where the specimen was cold-drawn 10% reduction before annealing at 650° C. for 6½ days. When annealed for 10 days, Fig. 25*d*, more graphite was observed. If the cold-drawing was increased to 30% reduction, more graphite after annealing at 650° C. was observed, Fig. 25*e*, but with further cold-drawing, say, 60% reduction, the amount of graphite observed after annealing was not necessarily more than with 30% reduction. In Fig. 25*f*, steel *F*1, cold-drawn 16% reduction, followed by subcritical annealing at 650° C. for 6½ days, the graphite nodules are more massive than in Figs. 25*c* and *e*. The investigations seem to indicate that graphitization in cold-worked steel on subcritical annealing increases directly with the carbon content of the steel.

The graphitized specimens were softer than those which had not been graphitized, but for obvious reasons the hardness figures could not be taken as a measure of the amount of graphitization. Even with micro-examinations, the results are only comparative because of the uneven distribution of the graphite. A more detailed study of graphitization has been carried out on cold-worked steel by the determination of colour-carbon, and it is hoped to report the results in the near furture.

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ANDREW CARNEGIE RESEARCH REPORT

# Section II—EFFECT OF COLD-WORK UPON ELECTRICAL RESISTIVITY IN STEEL

*With P. L. Chang, B.Sc., Ph.D., and R. Guenot, E.C.P.(Ing.), Ph.D.*


## SYNOPSIS

Electrical resistivity measurements have been extensively carried out on cold-drawn wires, both at room temperature and during or after subsequent heating, and the experimental methods are described in detail. On cold-drawing, the resistivity increases in the case of low-carbon steels or iron, but it decreases with high-carbon steels, as measured in the longitudinal direction of the wires. This decrease is greater if the carbon content of the steel is higher, and for a given steel the change of resistivity on cold-drawing is dependent upon the initial structure of the specimen.

On tempering, the resistivity of a cold-drawn low-carbon iron decreases continuously with increasing tempering temperature up to 550–600° C. With high-carbon steel wires, however, the change of resistivity on tempering is different, especially at low temperatures, where a small increase takes place over the range 150–450° C., with a maximum at about 250–300° C.

The effect of cementite distribution upon the resistivity of steel after cold-drawing has been investigated, and the lowering of resistivity in the longitudinal direction is shown to be less with a sorbitic than with a pearlitic structure. The experimental results are corroborated with the theoretical treatment.


THE measurement of electrical resistivity has been shown by many workers to be an invaluable method for the study of precipitation and solution processes, as well as order–disorder changes in metals and alloys. When the present investigation was first contemplated, it was considered that all these changes were possible and some might be involved when steels were cold-worked. This has led to the experiments described in this Section.

Two different approaches were made. The first was primarily concerned with the change of resistivity during heating or cooling, so that any difference between cold-worked and annealed specimens, either in temperature coefficient or in deviation in resistivity at any particular temperature, could be ascertained. The second approach was restricted to measurements at room temperature under standardized conditions. As will be shown later, although the two approaches were intended to have scopes of their own, in many cases the results are identical and can be used to check each other.

## MEASUREMENT OF RESISTIVITY AT ELEVATED TEMPERATURES DURING HEATING OR COOLING*

### EXPERIMENTAL TECHNIQUE

In experiments of this nature the tests must be carried out in vacuum to prevent oxidation of the specimen. Since the specimens are in the form of long wires, uniform heating over a considerable length presents certain difficulties. Not only must the method chosen and the arrangements made enable reproducible results to be obtained, but also a relatively high degree of accuracy is necessary to reveal any small changes associated with the effect of cold-work.

In the present experiments, wires 0·064 in. in dia. and about 8 in. overall in length were used. Such small wires are easier to draw than large bars, and high reductions can be reached with most steels; also, a small current gives a reasonably large potential drop, and a steady supply can be obtained from batteries over a considerable period. Further, with the differential method adopted in these experiments, as will be explained later, the smaller the size and the closer are the two specimens, the more uniform will be the temperature condition between them. The only disadvantage of a small specimen is the accuracy required to determine its diameter, since the error involved is proportionally large if the resistivity results are in absolute units. However, this does not affect the comparison between cold-worked and annealed specimens in the present case, since absolute measurements of the diameter were avoided and similar errors cancelled each other, as will be clear from the experimental procedure described later.

Measurements were made on a potentiometric principle, with a Tinsley vernier potentiometer incorporated in an electric circuit. Two specimens, one cold-worked and the other heat-treated, were placed side-by-side in a vacuum furnace and heated simultaneously. A 10-cm. length along the central part of each specimen was defined by two pairs of knife-edges from which the potential drop was tapped and measured.

#### Vacuum Furnace

As shown in Fig. 26, the furnace was in a vertical position and was supported on an iron frame. The furnace tube was 3 in. internal dia., and was wound with Nichrome wire along its 20-in. length. With care in winding and packing, the furnace gave a uniform temperature zone of not less than 5 in. along its centre. Inside the furnace tube was a silica tube, 2-in. bore and 30 in. long, with hollow brass water-coolers cemented to both ends, making flat ground joints with brass covers. Evacuation was carried out through the bottom cover by a Metrovac pump, which reduced the pressure inside the silica

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* The arrangements made and the results obtained in this part are the work of one of the authors (P.L.C.).

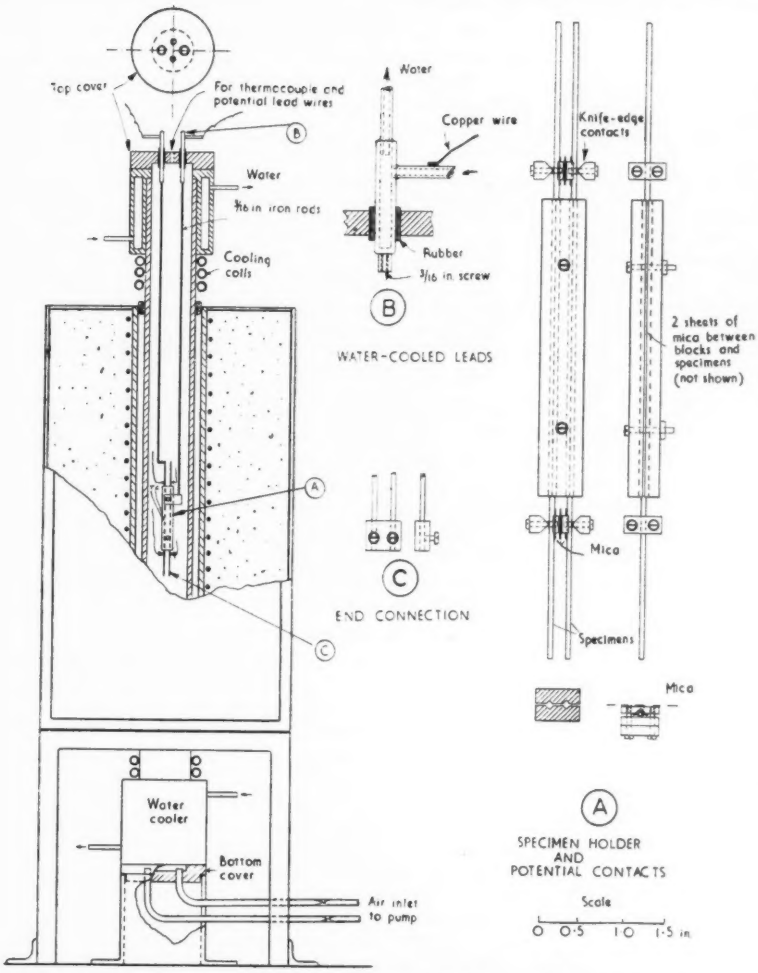



**Fig. 26—Arrangement of furnace, specimen holder, and potential contacts**

tube to less than 0·005 mm. Hg when the experiment was in progress. This prevented oxidation of the specimen even at the highest temperature (850–900° C.) in the experiments. The electrical leads and the thermocouple wires passed through the top cover, and were insulated from the brass either by silica sheathing or with a rubber cover and sealed with wax. The two main leads which supplied current and sustained the weight of the specimen and its holder, were $\frac{3}{16}$-in. dia. iron rods. To prevent softening of the sealing wax by conduction, a water-cooling device was employed at the top-cover joints for the two main leads, as shown in Fig. 26.

### Choice of Method of Measurement

Although the furnace gave a uniform temperature zone of at least 5 in., which was more than sufficient for the length of the specimen actually concerned in the measurement (10 cm. or about 4 in., *i.e.*, the distance between the two pairs of knife-edges), the problem of uniform heating and the measurement of the temperature of the specimen was not solved, particularly if information was to be derived from comparisons of temperature/resistance curves. As the temperature coefficient of resistance of iron is large, a temperature difference of, say, 3° C. between two experiments would produce a difference in resistivity of about 2%, which is nearly the amount of change due to the effect of cold-work. Thus, unless the temperature along the specimen was extremely uniform and the measurement was accurate to within 0·1° C., the study of the effect of cold-work, based on straightforward temperature/resistance curves would not be successful.

In view of this, a differential method, similar to that used by Stockdale,[24] in his studies of the phase boundaries of the Cu–Ag system, was employed. Using two specimens for each experiment, the difference between the two can be measured under identical conditions. For the present experiments the two specimens consisted of a cold-worked and an annealed specimen in each case, placed close to each other in a specimen holder (described below).

### Specimen Holder, Knife-Edges, and Electrical Contacts

The two specimens, identical in shape and size, were firmly clamped between two soft-iron **V**-blocks, $\frac{5}{8} \times \frac{3}{16} \times 3\frac{1}{2}$ in. (Fig. 26). Thin sheets of mica were inserted between the blocks and the specimens so that there was no electrical contact between them. The clamp fixed the distance between the two specimens at 0·22 in. centre-to-centre for all experiments, and it kept the specimens straight and reduced any temperature difference, point-to-point, between the two, thus ensuring identical conditions. This is particularly important, as already explained.

The knife-edge attachments were made of heat-resisting steel and are shown in Fig. 26. Four attachments were necessary and each consisted of a **V**-grooved guide and a knife-edge, fastened together by two small screws. The **V**-grooved guide was insulated from the specimen by mica. After the experiment the distance between the two marks made by the knife-edges was measured on a travelling vernier microscope, reading to 0·01 mm.

The two iron rods which supplied the current were screwed to the water-cooled leads at the top joints (Fig. 26) and were connected to the specimens by wedging into two separate iron blocks and fastening

with screws. The potential leads from the knife-edges were 26-S.W.G. soft-iron wires.

**Temperature Control and Measurement**

External resistances connected in series with the furnace winding were adjusted until the temperature inside the furnace was steady. This gave better control than a temperature regulator.

The temperature was measured by a Pt/Pt–10% Rh thermocouple connected to a separate potentiometer. The hot junction of this couple was inserted in a small hole drilled in the specimen holder at a position corresponding to the middle of both specimens. At times another thermocouple measured the temperature at the knife-edge contacts, and under steady conditions the difference between the knife-edge points and the centre of the specimens did not exceed 1° C., irrespective of the temperature. This difference was of no importance, provided that the temperatures of the two specimens, point-to-point, were the same.

**Electrical Circuits**

The electrical circuits are shown in Fig. 27. The current supply came from two batteries $AA$, connected in parallel. The two specimens $W_1$ and $W_2$, are in series with a standard resistance $R_s$ and a variable resistance $R_a$ ; $S_m$, $S_m'$, $S_1$, $S_2$, and $S_3$ are the reversing switches, and $G$ is the galvanometer. The resistances for adjusting the sensitivity of the galvanometer are shown by $r_1$ and $r_2$.

The total resistance of the electric circuit was about 2 ohms, and the current supply was 1 amp. As the temperature was increased, the current supply decreased slightly because of the increase in resistance. As is clear from the principle of the differential method, a slight variation in the current supply does not affect the results, and no attempt was made to maintain a constant current throughout the experiment.

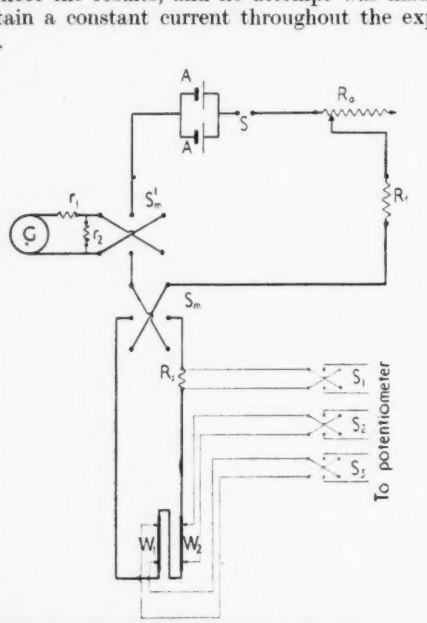



**Fig. 27—Circuit for resistance measurements**

**Operation**

When the required temperature was reached and had become steady, the D.C. supply from the batteries was switched on. After about 15 min. the current from the batteries became constant, and measurements were taken according to the following procedure :

(*a*) Observation of temperature
(*b*) Observation of potential drop of:
  (i) First specimen
  (ii) Second specimen
  (iii) Standard resistance
  (iv) Repeat (ii)
  (v) Repeat (i)
(*c*) The current was then reversed and readings as in (*b*) were recorded
(*d*) The current was reversed again and readings as in (*b*) were recorded
(*e*) Observation of temperature.

With practice this operation could be completed in 2 or 3 min., during which there was no change in the temperature of the specimens as indicated by measurements before and after the operation. The readings were averaged between (*b*) and (*d*), and these averaged readings were again averaged with (*c*). Thus, any effect associated with thermal e.m.f. or due possibly to slight temperature variations during the operation was cancelled or compensated.

**Preparation of Specimens**

It is not easy to machine long, thin specimens exactly to size. The annealed or otherwise heat-treated specimen representing the unworked state was therefore prepared from cold-drawn wires. To ensure similarity in initial structures, the following procedure was adopted. Blank rods, 0·25 in. dia., were machined from hot-rolled bars of ½-in. size and were subsequently cold-drawn to various diameters, for example, 0·125 in. They were then annealed or heat-treated at a temperature high enough to remove the effect of cold-work. After the specific treatments, they were again cold-drawn and finished at 0·064 in. dia. Two specimens, both 8 in. long, were cut from the cold-drawn wire ; one was preserved to represent the cold-worked state and the other was treated to the same initial structure as that prior to the second drawing. Annealing was carried out in a separate vacuum furnace and the temperature and time of soaking were as near as possible to those employed for the steel before the second drawing. If normalizing or isothermal treatment was desired, the specimen was enclosed in a copper tube, packed with turnings or filings of the same steel, and sealed at both ends, and then treated in the same way as before the second drawing. This specimen was taken as equivalent to the ' unworked ' state with respect to the corresponding cold-drawn specimen. The specimen was either annealed in vacuum or treated in an enclosed tube to prevent oxidation, thus preserving the original size and shape of the specimen. The very slight change in shape—a contraction in diameter (indicated in Section I to be associated with cold-drawn wires after subsequent heat-treatment)—was ignored. It should be pointed out here that, in this part, the term " annealed or heat-treated specimen " represents " unworked specimen " as specified above.

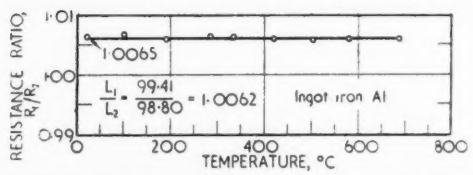



**Fig. 28—Resistance-ratio/temperature curve of two annealed specimens of ingot iron *A1*, to show experimental accuracy**

### Experimental Accuracy and Checking

In view of the many factors involved, it is difficult to give absolute accuracy. Checks were made by using two specimens of a given steel, heat-treated or annealed under identical conditions, and the ratio of their respective resistivities at different temperatures was measured. A typical ratio/temperature curve for *A1* is shown in Fig. 28. The ratio is constant at different temperatures, the scattering of the points being less than 0·1%. This indicates that ratio/temperature curves obtained with the present arrangements will be extremely sensitive to any small changes occurring in one specimen and not in the other. Also, the mean ratio in Fig. 28 is 1·0065 instead of 1·0000, owing to the difference in length of two specimens, the length ratio being 1·0062. When corrected, the deviation of the mean ratio from unity was not more than 0·03%.

## RESULTS

Results are available for a series of plain carbon steels, *A1* (0·04% C), *C1* (0·27% C), *D1* (0·58% C), and *E2* (0·71% C). In addition, a 3% Cr steel, *CR5*, has been studied in the $Fe_3C$ and the $Cr_7C_3$ conditions. The results can be expressed in several ways, and each is discussed in turn in the following pages.

### Temperature/Resistance Curves

For reasons already given, straightforward temperature/resistance curves are unsuitable for revealing small changes associated with the effect of cold-work upon iron and steel. These curves, although available, are not reproduced here.

*Reversion of Cementite to Chromium Carbide on Continuous Heating*—The temperature/resistance curves for steel *CR5*, isothermally treated at 700° C. to give $Cr_7C_3$, or at 350° C. to give $Fe_3C$, are shown in Fig. 29. The specimen treated at 350° C. showed a higher resistance than that of the same steel transformed at 700° C., the difference in resistivity at room temperature being about 20%. In Fig. 29 the two curves are nearly parallel up to about 500° C., but above this they converge and eventually become identical at about 650° C.

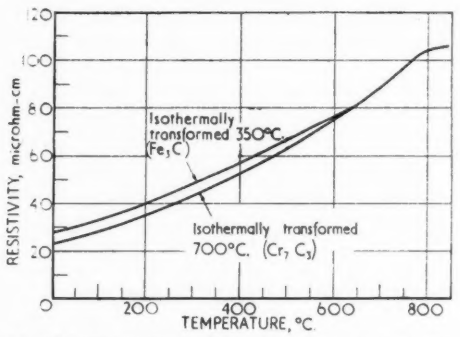



**Fig. 29—Temperature/resistance curves of steel *CR5* in isothermally transformed conditions**

Ignoring the difference in size and distribution of the carbides in these two specimens, it seems that, since isothermal treatment at 350° C. gives rise to cementite, most of the chromium would be in solid solution with the ferrite ; therefore the resistivity of such a specimen might be expected to be higher than that of the specimen isothermally transformed at 700° C., in which most of the chromium would be taken up by formation of $Cr_7C_3$. Crafts and Lamont[25] have shown that with long-time tempering the reversion of cementite into chromium carbide in quenched chromium steels could be accomplished only at a temperature not much lower than 500° C. This would seem to fit in with the temperature/resistance curves in Fig. 29. It is possible that, with the particular rate of heating in the present experiments, the reversion of cementite to chromium carbide commences at about 500° C., but it may not be complete until 650° C.

### Resistivity-Ratio Temperature Curves

The differential method directly gives the resistance ratio between cold-drawn and annealed specimens at different temperatures. After correcting any difference in the length of the specimen, the results can be expressed as resistivity ratios, which are plotted against the temperature in Fig. 30 for specimens *A1*, *C1*, *D1*, *E2*, *CR5* ($Cr_7C_3$), and *CR5* ($Fe_3C$). In this series all the cold-drawn specimens had received 73·5% reduction. The four plain carbon steels were annealed, and the designations $Cr_7C_3$ and $Fe_3C$ for *CR5* correspond with the isothermal treatment at 700° C. or at 350° C., respectively, before cold-drawing.

In carrying out the experiments, the resistance ratio of the cold-drawn and the corresponding ' unworked ' specimen was first measured at room temperature and at successively higher temperatures. At certain temperatures the specimens were allowed to cool and the ratio was followed to room temperature during cooling. These cooling curves are shown as broken lines in Fig. 30. When the specimens were reheated and the ratio at the different temperatures was again measured, it followed the broken curve until the temperature from which the specimens were first cooled was reached, after which the ratio proceeded along the full-line curve. Thus, the full-line curve represents the relation between the resistivity ratio and the temperature without subjecting the two specimens to intermediate cooling and reheating. This applies to all the steels examined.

The ratio curves, as shown, are the intermediate step for obtaining the difference ($\Delta R_t$) curves given below.

### $\Delta R$/Temperature or $\Delta R_t$ Curves

Let $\Delta R = R' - R$ be the resistance difference at room temperature between two specimens of identical

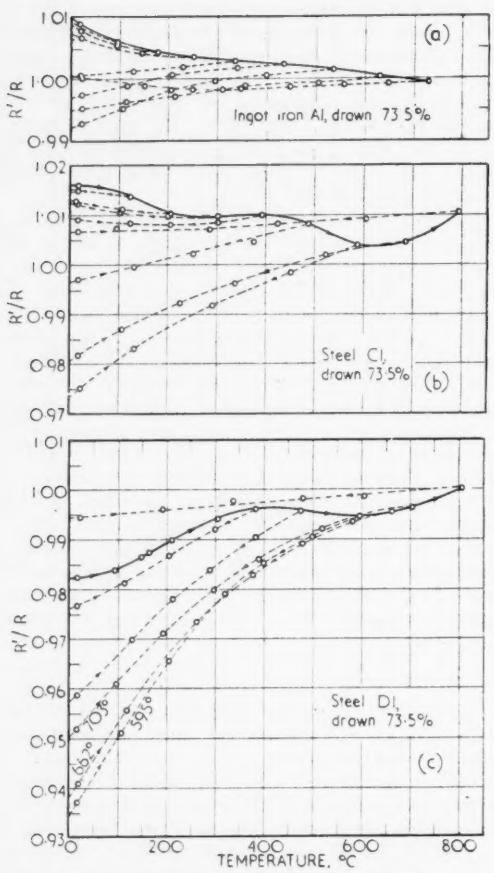



**Fig. 30—Resistance-ratio/temperature curves for cold-drawn specimens (73·5% reduction)**

size, and if no change takes place during heating, the difference should remain constant at all temperatures, as in Fig. 31, curve (*a*). If changes happen to occur, the $\Delta R_t$ curve will be quite different. Consider a simple case in which changes occur in one and not in the other during heating; the modification of the $\Delta R_t$ curve will mainly depend upon the nature of such changes. Thus, if two separate changes take place at two temperatures, say, at $T_A$ and $T_A'$, there will be two discontinuities corresponding to the two changes, as in Fig. 31, curve (*b*). If, however, the changes happen to be a gradual process which proceeds

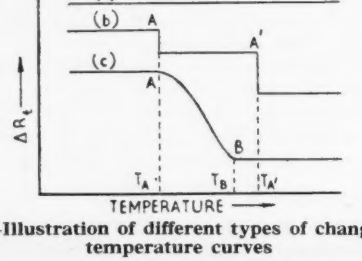
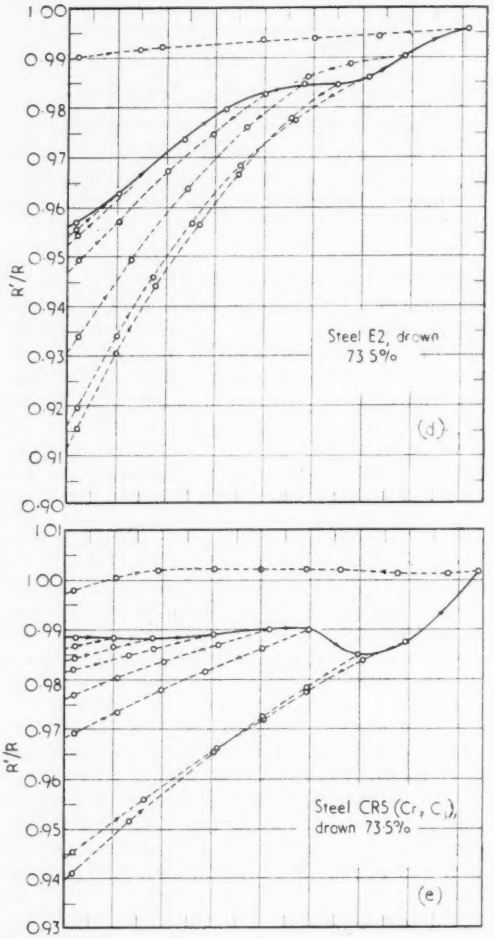



**Fig. 31—Illustration of different types of change in $R_t$/temperature curves**



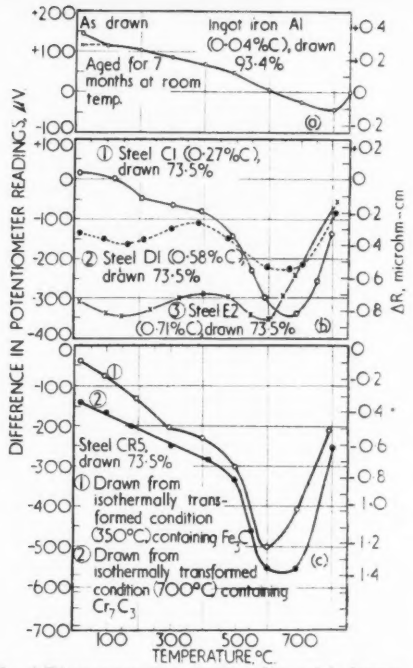

As drawn
Ingot iron A1 (0·04%C), drawn 93·4%
Aged for 7 months at room temp.
(a)
① Steel C1 (0·27%C), drawn 73·5%
② Steel D1 (0·58%C), drawn 73·5%
③ Steel E2 (0·71%C), drawn 73·5%
(b)
Steel CR5, drawn 73·5%
① Drawn from isothermally transformed condition (350°C.) containing $Fe_3C$
② Drawn from isothermally transformed condition (700°C.) containing $Cr_7C_3$
(c)
DIFFERENCE IN POTENTIOMETER READINGS, μV
ΔR, microhm-cm
TEMPERATURE °C.

**Fig. 32—$\Delta R$/temperature curves for cold-drawn specimens**

as a function of temperature between $T_A$ and $T_B$, then the $\Delta R_t$ curve may assume the shape of curve (*c*).

Having made these conceptions clear, we may proceed to describe the methods by which the $\Delta R_t$ curves can be derived from experimental results. Since $\Delta R = R' - R = (R'/R - 1)R$, where $R'/R$ is the ratio and $R$ is the resistance of the unworked specimen, both values are known from the resistivity-ratio/temperature and the temperature/resistance curves, respectively, and $\Delta R$ can therefore be calculated for all temperatures. Alternatively, $\Delta R_t$, which represents the values of $\Delta R$ at different temperatures, can be obtained directly from the potential drop measured for the two specimens under different temperature conditions. In the latter case, the results are expressed in terms of potentiometer readings after correcting for any difference in the length of the two specimens. As expected, the results were identical for both methods.

The $\Delta R_t$ curves thus obtained for *A*1, *C*1, *D*1, *E*2, *CR*5 ($Cr_7C_3$), and *CR*5 ($Fe_3C$) are shown in Fig. 32. With the exception of *A*1, which was cold-drawn 93·4%, the reductions for the other steels were similar, about 73·5%. In these graphs, the values for $\Delta R$ are given in microhm-centimetres, as well as in difference in potential-drop readings corresponding to a current supply of 0·85 amp. and a specimen length of 10 cm. A comparison between these two units will give an idea of the magnitude of the relative quantities concerned in the measurements. The values given in terms of the absolute unit (microhm-cm.) may be only approximate because of the relatively large percentage error involved in the measurement of the small diameters.

As shown in Fig. 32*a*, the resistance difference ($\Delta R$) between the cold-drawn and the unworked specimen decreases slowly with increase of temperature from room temperature to just below 500° C. Above this temperature, the curve follows a steeper course until 700° C., where the decrease slows down and finally the curve actually turns upward, showing a minimum at about 800° C. Ignoring the cause of the last change for the time being, it is suggested that the decrease in $\Delta R$ below 500° C., which appears to be a continuous process, may be mainly associated with the release of internal strains in the cold-worked ferrite. Above 500° C. the more rapid decrease is thought to be connected with recrystallization and grain growth.

The results for the three carbon steels, *C*1, *D*1, and *E*2 (Fig. 32*b*) seem to be more complicated. First, the resistance difference ($\Delta R$) between the cold-drawn and the unworked specimen, measured at room temperature, was actually smaller for *C*1 than for *A*1 (*cf.* Fig. 32*b*, curve (1), with Fig. 32*a*). With the two high-carbon steels (*D*1 and *E*2) the resistance difference at room temperature was negative (Fig. 32*b*, curves (2) and (3)), which means that the resistance of the cold-drawn specimen was actually less than that of the unworked one. This has been attributed to the effect of carbide distribution in steel, as will be explained later.

In all three curves in Fig. 32*b*, $\Delta R$ decreased with increasing temperature at the initial stage of heating. At 150–200° C. this decrease was halted, and with the two high-carbon steels (*D*1 and *E*2) $\Delta R$ increased from 150° to about 400° C. (Fig. 32*b*, curves (2) and (3)). Above 500° C., $\Delta R$ decreased again, but more rapidly than during the initial stage of heating, and this continued until 600–700° C., after which the curves rise sharply well into the $\gamma$ range.

The decrease in $\Delta R$ on heating is to be expected on the basis of the release of internal strains in cold-worked specimens. With the ingot iron *A*1, this continues up to 500° C. Clearly, the same thing must happen in carbon steels. If this contention is correct, it appears that the discontinuity between 150° and 400° C., as shown in Fig. 32*b* for the three carbon steels, must be due to another process which contributes to an increase in the resistance of the cold-worked specimens over and above the effect associated with the release of internal strains. On this basis, it seems that since the process increases in intensity with increasing carbon content of the steel, it may be connected with certain changes taking place in the cementite when cold-worked specimens are subjected to heating. The results on *CR*5 may now be quoted :

Figure 32*c*, curve (2), shows the $\Delta R_t$ curve for *CR*5 in the $Cr_7C_3$ condition : $\Delta R$ decreases with increase of temperature and there is no discontinuity of the curve between room temperature and 500° C. When the same steel was cold-drawn in the $Fe_3C$ condition, however (curve (1)), the $\Delta R_t$ curve shows a discontinuity at 300° C., which is very similar to that reported for *C*1 at about 200° C.

Above 500° C. the curves for all the steels are similar, and they all show a minimum between 600° and 700° C., the exact temperature depending upon the composition and treatment of the steel. This minimum can be compared with that observed for the ingot iron *A*1 at 800° C. Between 500° C. and

the minimum, the decrease in $\Delta R$ is attributed to recrystallization and grain growth. Above the minimum, the rapid increase in $\Delta R$ in the steel specimens is mainly associated with a change from fibre to random structures. This point, and the slight increase in $\Delta R$ above 800° C. in the ingot iron (Fig 32*a*) will be discussed later. In the meantime, it should be noted, in so far as electrical resistance measurements are concerned, that cold-drawn steels cannot be completely restored to the unworked state unless they are subsequently annealed at a temperature well above the $\alpha$–$\gamma$ change point so that $\Delta R$ is zero and remains so when the annealing temperature is increased further.

**Resistance Ratio between Cold-Drawn and Unworked Specimens at Room Temperature**

From the resistance-ratio/temperature curves in Fig 30, values of the resistance ratio at room temperature between cold-drawn and unworked specimens after tempering at different temperatures can be obtained. Since, in most cases, the resistance of the unworked specimen was unaffected by tempering, it is thought that the resistance ratio, as measured at room temperature, plotted against the tempering temperature, should provide information on the effect of tempering upon the change of resistance in cold-worked steels. The only exception would be *CR*5 in the $Fe_3C$ condition, because the resistance of the unworked specimen would not remain constant after tempering at different temperatures, especially above 350° C., the temperature of isothermal treatment before cold-drawing. Even so, the results should give an idea of the removal of cold-working effect after tempering, provided that the normal constitutional changes during heating, such as the reversion from cementite to chromium carbide, are similar for cold-drawn and unworked specimens.

In Fig. 33*a* two curves are shown for the ingot iron *A*1, curve (1) for a specimen of 93·4% reduction and curve (2) for 73·5% reduction. In curve (1) the full line was obtained soon after drawing, and the broken line, which coincides with the full line above 100° C., was based on measurements carried out on a similar specimen which had been aged at room temperature for seven months. In the freshly drawn condition the 93·4% reduction specimen showed a resistance 3% higher than that of the unworked specimen at room temperature. With the aged specimen, however, this difference was smaller, about 2·5%. The rapid decrease in resistance between room temperature and 100° C., as shown by the full line, must therefore be due mainly to accelerated ageing. This phenomenon has not been studied for the 73·5% reduction specimen, and curve (2) was obtained after the specimen had been fully aged.

Restricting the remarks only to fully aged specimens, the variation of the resistance with tempering temperature is very similar for the two cold-drawn specimens (93·4% and 73·5% reduction, curves (1) and (2) in Fig. 33*a*). Thus, both specimens showed a minimum resistance after tempering at 550° C. and this minimum was about 0·8% below that of the unworked specimen in each case, in spite of the fact that in the as-drawn condition the resistance

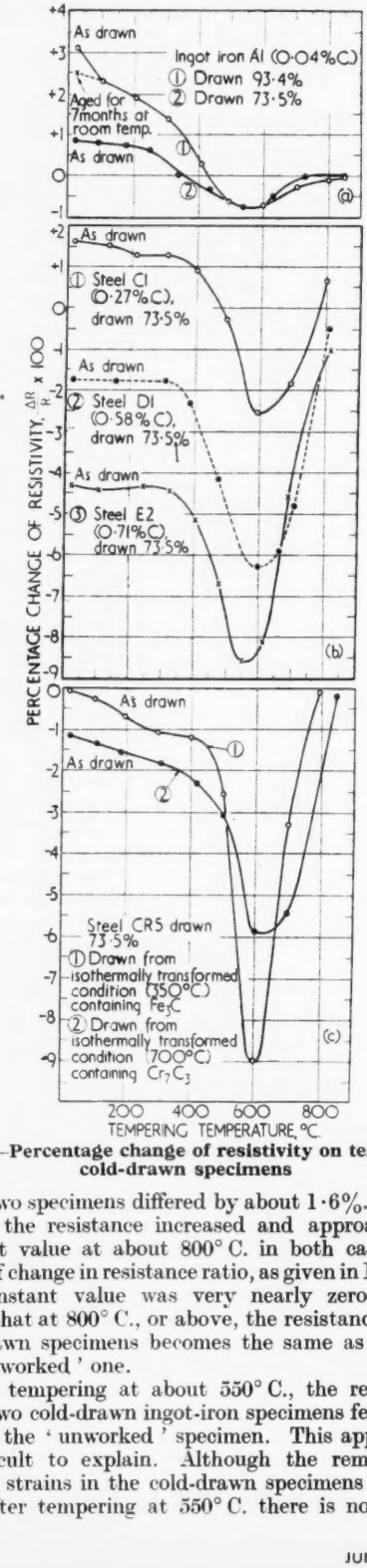



**Fig. 33—Percentage change of resistivity on tempering cold-drawn specimens**

of the two specimens differed by about 1·6%. Above 550° C. the resistance increased and approached a constant value at about 800° C. in both cases. In terms of change in resistance ratio, as given in Fig. 33*a*, this constant value was very nearly zero, which means that at 800° C., or above, the resistance of the cold-drawn specimens becomes the same as that of the 'unworked' one.

After tempering at about 550° C., the resistance of the two cold-drawn ingot-iron specimens fell below that of the 'unworked' specimen. This appears to be difficult to explain. Although the removal of internal strains in the cold-drawn specimens is complete after tempering at 550° C. there is no reason

to suppose that the resistance of such specimens should be lower than that of the unworked one. On the contrary, the grain size of the cold-drawn specimens recrystallized at 550° C. would be smaller than that of the unworked one in these experiments and, as shown later, small-grained specimens had slightly higher resistance at room temperature than large-grained ones.

The $\Delta R_t$ curve of this ingot iron also showed a minimum (Fig. 32*a*), but at 800° C. instead of 550° C. It is likely that the two minima may be connected with an early precipitation process, the mechanism of which is obscure. However, some previous workers, for example, Edwards,[26] suggested that, even after annealing, the ingot iron is supersaturated with respect to carbon, nitrogen, or oxygen, and the precipitation of phases containing such elements on cold-working and ageing will presumably lower the resistivity of the ferrite. The re-solution of such phases on reheating may be responsible for the increase of resistivity above 800° C. in Fig. 32*a* and above 550° C. in Fig. 33*a*. On this assumption it may be significant that the decrease of resistivity on ageing, and the difference in resistivity between the specimen annealed at 550° C. and the unworked one, are of the same order, 0·5–0·8% (*see* Fig. 33*a*, curve (1)).

Curves for the plain carbon steels, *C*1, *D*1, and *E*2, are shown in Fig. 33*b*. No ageing effect was observed in these specimens. With *C*1 (curve (1)), resistance of the cold-drawn specimen (73·5% reduction) decreased slightly after tempering at 200° C. and then remained constant up to 300° C. With the two high-carbon steels *D*1 and *E*2 (curves (2) and (3)), the resistance of the cold-drawn specimens (both 73·5% reduction) was apparently unchanged up to 300° C. As mentioned before, the change of resistance in cold-worked carbon steels on tempering up to 400° C. may involve two processes, *viz.*, the release of internal strains, which must reduce the resistance, and a certain change associated with the cementite, which appears to increase it. These two processes overlap, and their effects upon resistivity oppose each other. Depending on the carbon content of the steel and the type of carbide present, the change in the resistance of a cold-drawn specimen after tempering, especially below 400° C., will vary.

This is further borne out by the results on *CR*5 in Fig. 33*c*. Curve (2) shows the results for *CR*5 in the $Cr_7C_3$ condition. The change in the resistance of a cold-drawn specimen (73·5% reduction) on tempering was gradual, and the manner in which the resistance decreases below 400° C. was very similar to that observed with an ingot iron. When the same steel was cold-drawn in the $Fe_3C$ condition, however, the curve for a 73·5% reduction specimen, curve (1), indicates an inflexion at 250–300° C. From there onwards the resistance was practically constant up to 400° C. This phenomenon is not unlike that observed with the plain carbon steel *C*1.

Above 400° C. the resistance of the cold-drawn specimens of all the steels examined decreased very rapidly. Depending on the composition of the steel, a minimum resistance was obtained usually after tempering between 550° and 650° C. In all cases, this minimum resistance was considerably smaller than that of the unworked specimen. On tempering at a still higher temperature, the resistance of all the specimens began to increase. After heating to 800° C., or slightly above, the tendency was for the cold-worked specimens to become fully annealed and to reach a state similar to that of the unworked ; in such a case the percentage change of resistivity as given in Figs. 33*b* and *c* would be zero.

It should be realized that, with steels, the resistance of a cold-drawn specimen is not governed by internal strains only. Thus, the elongated structure of alternate layers of cementite and ferrite as produced by cold-drawing must give rise to a lower resistance in the longitudinal direction of the wire than if the cementite particles or lamellae are distributed at random throughout the mass. The effect of this upon resistivity is opposite to that caused by internal strains which increase the resistance. The resistance of a cold-drawn steel measured at room temperature represents, therefore, the aggregation of these two opposite effects. Having made this point clear, it will be of interest to consider some of the results summarized in the following table :

*Change of Resistance in Steels at Room Temperature in the As-Drawn Condition and after Tempering to Minimum Resistance*

| Steels | As-Drawn (73·5% Reduction), % | Minimum Resistance, % |
|---|---|---|
| *A*1 (0·04% C) | +0·9 | −0·8 (T. 550° C.) |
| *C*1 (0·27% C) | −1·6 | −2·6 (T. 625° C.) |
| *D*1 (0·58% C) | −1·8 | −6·3 (T. 600° C.) |
| *E*2 (0·71% C) | −4·3 | −8·6 (T. 550° C.) |
| *CR*5 ($Cr_7C_3$) | −1·2 | −6·0 (T. 650° C.) |
| *CR*5 ($Fe_3C$) | −0·1 | −9·1 (T. 600° C.) |

The negative sign in these results means that the resistance of the cold-drawn specimen is smaller than that of the unworked, and the reverse is true when positive signs are indicated. In all cases, the difference is given in terms of percentage resistance of the unworked specimen. Considering the change of resistance in carbon steels only, it will be seen from the table that, after tempering the cold-drawn specimens at 550–625° C., a minimum resistance was reached. In such a case, the difference in resistance between the cold-drawn and the unworked specimen was at a maximum, and this difference became greater when the carbon content of the steel was higher. This observation is rather significant, because after tempering at such temperatures as 550–625° C., the removal of internal strains from the cold-drawn specimens may be regarded as complete ; the only factor which remains unchanged is the elongated structures, as shown by Fig. 24 of Section I. Ignoring the small effect due to the difference in grain size between the unworked and the cold-drawn and tempered specimens, the part played by the elongated structures in lowering the resistance of the cold-drawn specimens may therefore be estimated. These values are 2·6, 6·3, and 8·6% for steels *C*1, *D*1, and *E*2, respectively, as given in the above table.

The increase in resistance after the minimum, as shown in Fig. 33*b*, can now be explained. As shown by Fig. 24, the elongated structures of cold-

drawn specimens were gradually destroyed and changed into random distribution when the heating temperatures increased above 600° C. In such a case the difference in structure between the unworked and the cold-drawn specimens became smaller and smaller until a temperature was reached where both specimens were fully annealed and their structures became identical. At this stage the difference in resistance between the two specimens would be zero.

Referring to the foregoing table, the change of resistance due to cold-drawing (73·5% reduction), as measured at room temperature, was negative for *D*1 and *E*2, but positive for *A*1 and *C*1. This could only mean that with the two high-carbon steels the effect of elongated structures produced by drawing lowers the resistivity of the specimen more than the effect of internal strains increases it. When the carbon content of the steel is low and the amount of cementite present is proportionally small, as in *C*1, the reverse in the balance of these two opposite effects might be expected.

The difference in the values obtained between as-drawn specimens and those tempered to a minimum resistance, as shown in the table, can be taken as a measure of the internal strains in the different steels. Thus, for the ingot iron *A*1, this difference was 1·7%. For the carbon steels *C*1, *D*1, and *E*2, the corresponding differences were nearly the same, 4·2, 4·5, and 4·3%, respectively. This suggests that, for a given reduction, the internal strains in cold-worked steels are greater than in cold-worked iron. This deduction is in agreement with the X-ray results described in a later Section.

With regard to the results on *CR*5, it is not always possible to ascertain the effect of internal strains upon the resistance of cold-drawn specimens, as with plain carbon steels. For example, when the steel was treated and cold-drawn in the $Fe_3C$ condition, the removal of the internal strains on tempering was accompanied by a change of the carbide constitution above 500° C. This complication does not, however, apply to specimens initially treated to the $Cr_7C_3$ state, and the results in the table seem to indicate that, for a given reduction, the internal strains present in cold-drawn specimens are of the same order for the chromium steel as for plain carbon steels.

The effect of elongated structure or carbide distribution upon resistivity appeared to be even greater in chromium than in plain carbon steels. The reason for this may be that, for a given carbon content, the amount would be greater if the carbides are present as chromium carbide ($Cr_7C_3$) than as cementite ($Fe_3C$). Moreover, if the electrical resistivity of chromium carbide is higher than that of cementite, the effect of distribution will be further enhanced. Precise information regarding this latter point, however, is lacking.

#### Temperature Coefficient of Resistance of Cold-Drawn Specimens after Tempering at Different Temperatures

On cooling cold-drawn specimens from various tempering temperatures, the change of resistance follows the broken lines in Fig. 30, which also apply for reheating. This indicates that the temperature coefficient of resistance of cold-drawn specimens was modified by tempering and that it varied with the temperature.

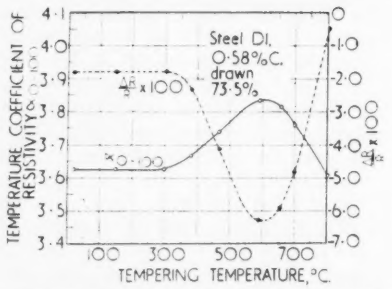



**Fig. 34—Effect of tempering on the temperature coefficient of resistivity of cold-drawn steel *D*1**

The temperature coefficient of resistance is defined by :

$$\alpha_{0-100} = \frac{R_{100} - R_0}{R_0} \times \frac{1}{100}$$

where $\alpha_{0-100}$ is the coefficient between 0° and 100° C., and $R_{100}$ and $R_0$ are the resistance at 100° and 0° C., respectively. Using these notations to represent the different quantities for the unworked specimen and $\alpha'_{0-100}$, $R'_{100}$, and $R'_0$ for the cold-drawn wire, it will be seen that :

$$\alpha'_{0-100} = \frac{R'_{100} - R'_0}{R'_0} \times \frac{1}{100} = \left(\frac{R'_{100}}{R_{100}} \times \frac{R_{100}}{R_0} \times \frac{R_0}{R'_0} - 1\right) \times \frac{1}{100}$$

Since $R'_{100}/R_{100}$ and $R'_0/R_0$ can be obtained from the resistivity-ratio/temperature curves and $R_{100}/R_0$ from the temperature/resistance curves, $\alpha'_{0-100}$ can be calculated. The results for *D*1 are given in Fig. 34. The change of temperature coefficient on tempering was the reverse of the change in resistance (for this comparison curve (2) of Fig. 33*b* is replotted as a broken line). This relation applied to all other steels examined.

## MEASUREMENT OF RESISTIVITY AT ROOM TEMPERATURE*

### EXPERIMENTAL TECHNIQUE

#### Preliminary Considerations

In the previous part, the change of resistivity in cold-drawn specimens during or after heating was determined by a differential method in which the cold-drawn specimen was referred to a standard, heated under identical conditions. The standard specimen, which represented the unworked state, was prepared from cold-drawn wires by annealing. When it was realized that electrical resistivity in steel depends to some extent upon the state of division and distribution of the carbides present, it was considered desirable to study the effect of cold-drawing upon specimens of widely different initial structures, for example, spheroidized sorbite and lamellar pearlite.

* The arrangements made and the results obtained in this part are the work of one of the authors (R.G.).

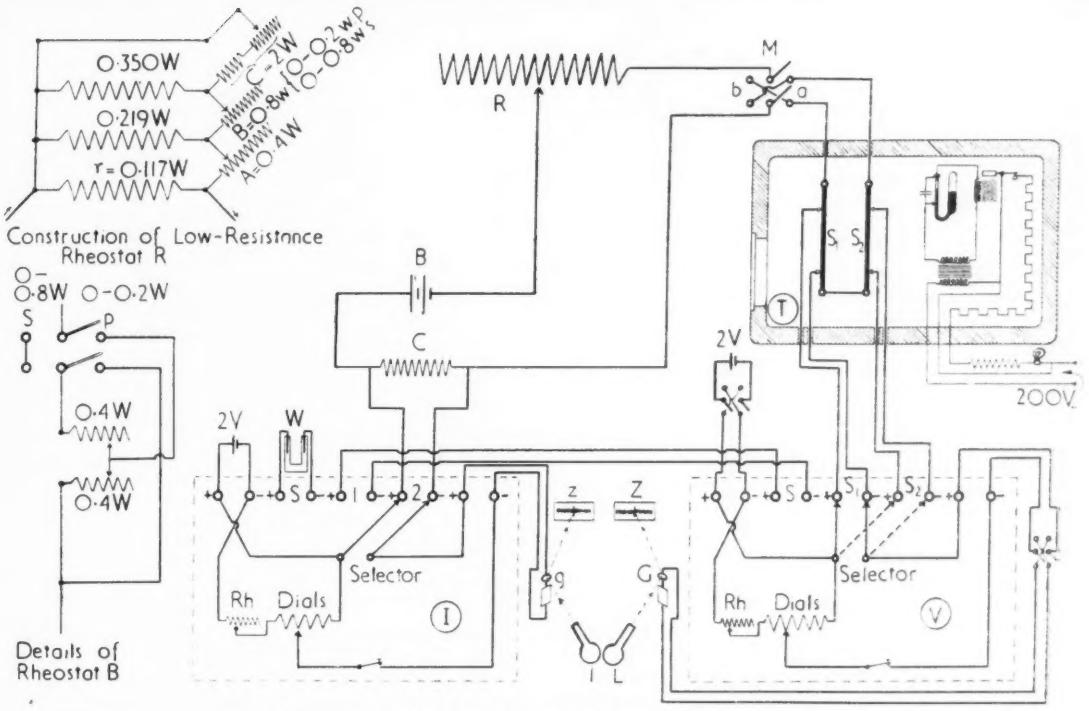



**Fig. 35—Layout of the arrangements for resistivity measurements**

The present investigation was planned to carry out measurements at room temperature in which the resistivity was directly compared with cold-worked and unworked specimens. To prepare the unworked specimen from bars of various initial treatment, a relatively large finishing diameter was necessary for accurate machining. Specimens of $\frac{1}{4}$ in. dia. and 8–10 in. overall in length were finally adopted, the effective length, *i.e.*, the distance between the two potential contacts, being 10 cm. in all cases. The current supply was increased proportionally to 25–30 amp.

The total resistance of the measured portion of this steel specimen was only about 500 microhms. Since the change of resistivity on cold-working and on subsequent tempering was not expected to be more than a very small fraction of this, it was essential for the method of measurement to give a high degree of accuracy. The method adopted was based on a differential principle, and the measurement was carried out by means of a Tinsley vernier potentiometer. At first, it would seem that, with the large specimen ($\frac{1}{4}$ in. dia.) and the high current (25–30 amp.), the Kelvin bridge principle might be the most suitable, as the results are independent of current fluctuations. On the other hand, such methods require some current to be passed through the potential contacts, even when the balance is reached, and the contact resistances may become a source of error. In the present case, sharp razor blades were used for potential contacts so that the length of the measured portion could be accurately defined and the resistance of the contacts was relatively high, about 1 ohm. Although the effect of contact resistances could be minimized by increasing the resistance in the galvanometer circuit, it would jeopardize the sensitivity of the measurement.

The potentiometric method has completely overcome the effect of contact resistances, as will be clear from the principle outlined below. By measuring successively the potential drop across the specimen and a standard resistance when the same current is flowing, the ratio of the potential drop is equal to the ratio of the resistances. Once the potentiometric balance has been reached, no current flows through the potential contacts and therefore the contact resistances may be relatively high without causing any inconvenience.

The high current (25–30 amp.) necessary for accurate measurements has given rise to certain difficulties, *viz.*, the heating of the specimen during measurements and the fluctuation of the current supply. To overcome these the apparatus was constructed on a differential principle in which two specimens of very nearly the same resistance were compared under conditions for which errors due to these two main sources could be compensated, as described below.

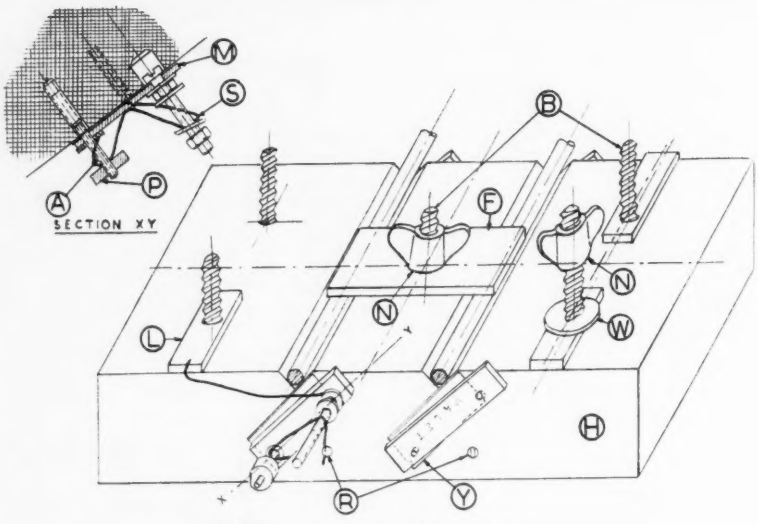
### Construction of the Apparatus

The layout of the arrangement is shown in Fig. 35. The current was supplied by two large batteries $B$, of low internal resistance, connected in series to give 4 V. The two specimens, $S_1$ and $S_2$, also connected in series, were placed in a specimen holder with razor blades for potential contacts. The specimen holder was immersed in an oil bath enclosed in a thermo-

statically controlled and heat-insulated box $T$. The specimens were connected to the batteries through a mercury reversing switch $M$, and two external resistances $R$ (a rheostat) and $C$ (a manganin standard, 0·005 ohm., of the four-terminal type, used primarily for calibration).

The measuring circuit incorporated two Tinsley vernier potentiometers, $I$, connected to the potential terminals of the manganin standard to measure the current, and $V$, for comparing the voltage drops across the knife-edges attached to the two specimens. The connections could be changed from one specimen to the other by the selection switch in the potentiometer.

One Weston cell $W$ was used for calibrating both potentiometers. It was always connected to potentiometer $I$, and the standard cell terminals of potentiometer $V$ were connected to one pair of terminals in potentiometer $I$ for mutual standardization. Two galvanometers ($G$ and $g$), lamps ($L$ and $l$), and scales ($Z$ and $z$) were used. The two scales were fitted with adjustable zeros and were placed one above the other for quick observation.




*H* Holder
*F* Fastening spring
*W* Washer
*L* Connecting lug
*M* Blade mounting
*N* Wing nut
*A* Rotating axis
*S* Spring
*P* Pressure screw
*B* Brass screw
*Y* Razor blade
*R* Spring rest

**Fig. 36—Details of specimen holder**

*Specimen Holder and Knife-Edges*—These are shown in Fig. 36. The two specimens were placed side-by-side in two separate **V**-grooves in a bakelite block, and were clamped down under a flat piece of bakelite by a wing nut. Two razor blades were mounted on each side of the block, the distance between the two blades on each specimen being 10 cm. As shown in Fig. 36, each blade was mounted rigidly on a copper flat and held against the side of the block by a hard spring. This prevented side play, but did not interfere with the free movement of the blade which could slide about $\frac{1}{8}$ in. in its own plane. By adjusting the compression on the spring by means of a fixed screw, the sharp edge of the blade exerted a slight pressure against the specimen.

*Current Terminals and Specimen-Ends*—The construction is shown in Fig. 37. Both ends of the specimens were slightly tapered to fit with part of the sockets in two identical bakelite blocks. The remainder of the sockets were filled with mercury. A copper plug was permanently force-fitted into each end of the specimens and finally amalgamated to ensure excellent contact with mercury.

The specimens are easily replaced, but the main purpose is to reduce the contact resistance to a minimum and to render it as consistent as possible to prevent heat being generated at the junction when a high current is flowing. This localized heating would result in a temperature gradient along the bar and might cause a temperature difference between the two specimens, thus altering the resistivity ratio. This was experienced with earlier designs of current connection, where relatively high and inconsistent contact resistances caused an unsteady voltage drop, a high e.m.f. at the potential junctions, and a rapid change in the temperature conditions of the specimens, so that the same reading could not be repeated at intervals.

In the authors' experience, a high thermal e.m.f. at the potential contacts indicates poor current connections. It also constitutes a direct source of error because such an e.m.f. is never steady enough to be completely cancelled by reversing. With the present design, the e.m.f. encountered during measurements was usually very low and was kept below 4 $\mu$V., or if this value was exceeded, the connections were overhauled and the cause was investigated before measurements were taken.

*Current Supply and Regulation*—As already mentioned, a current of 25–30 amp. was used, and was supplied by two low-resistance accumulators in series to give 4 V. and a capacity of about 150 amp.hr. The total resistance of the circuit was only about 0·15 ohm, and effective stabilization and regulation of the current were difficult.

In the present experiments the ballast resistance to regulate the current was limited to less than 0·1 ohm, and care had to be taken to reduce contact resistances. Slight local heating in any part of the circuit could cause instability of current supply. To overcome this, temporary contacts, such as switches or sliding rheostats, were avoided as far as possible, and leads and connections of 500-amp. normal rating carried the current. The only switch which could not be avoided was the mercury reversing switch $M$ (Fig. 35), with an 'off' position.

The ballast resistance $R$ (Fig. 35) consisted of a

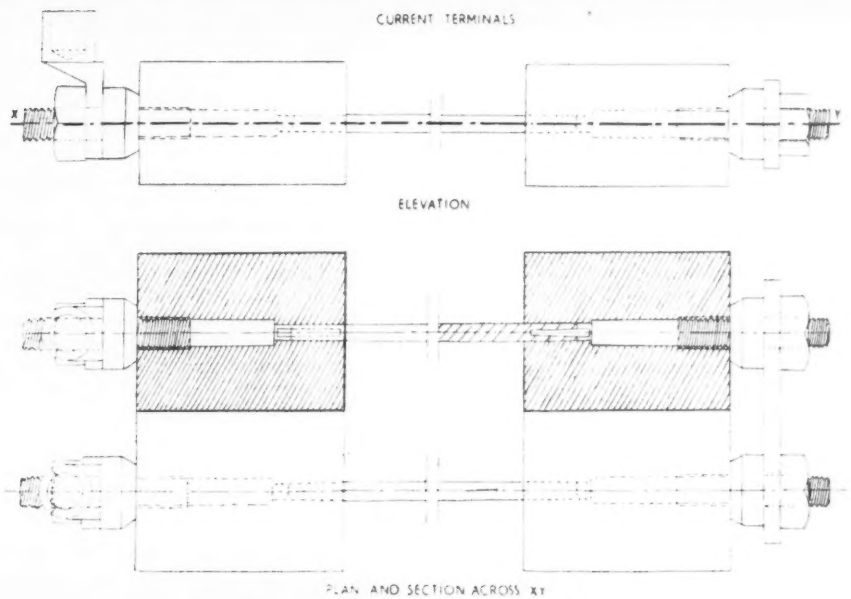



**Fig. 37—Construction of current terminals**

circuit with a total resistance of about 0·1 ohm. The main resistor $r$, which was made of thin copper strips suspended in cold water, took about four-fifths of the total current. The remainder passed through the shunt circuits which were designed for the necessary adjustment. As shown in Fig. 35, the rheostats $A$, $B$, and $C$ are calculated to give sensitivities of adjustment in the proportions of 1, 10, and 100, respectively. In these auxiliary circuits the total resistances were relatively high, and sliding rheostats could be used to facilitate operation. The present arrangement includes two overlapping ranges corresponding to the positions $s$ and $p$, to cover the required variations in e.m.f., and gives a fineness of adjustment of the order of 1 part in 100,000.

*Temperature Control*—The whole arrangement for resistivity measurements was situated in a small room maintained at 22° C. by a toluene-bulb controller operating a 2-kW. radiator. This reduced the variation of the room temperature to within ± 0·1° C. The specimen holder was placed in a paraffin oil bath enclosed in a wooden box with an internal capacity of about 1 cu. yard. The outside of the box was covered with thick felt to assist heat insulation. Inside the box, fine resistance wires were wound along the four walls ; the windings were non-inductive and regularly spaced, with a total resistance of about 200 ohms. A current of 0·2 amp. was passed through the windings and was switched on and off by another toluene-bulb controller inside the box. This current maintained the same temperature inside the box throughout the seasons because the outside temperature was controlled at the same level. When steady conditions were reached, the temperature of the oil bath did not vary by more than 0·01° C., as measured at regular intervals by a platinum resistance thermometer. The outside control also ensured an approximate constant temperature for the standard resistance and cells. With the specimens immersed in the oil bath, any temperature variation due to oscillation of the thermostat was further smoothed out and also facilitated heat dissipation from the specimens when current was flowing.

### Operation

*Preparation of Specimens*—Two specimens, ¼ in. dia. and a suitable length, were used each time. For cold-drawn specimens, heat-treated bars of different sizes were reduced in successive steps and were finished through a ¼-in. die to give different reductions. The unworked specimen was prepared by machining alone, but often a light draft through the ¼-in. die was necessary to obtain the desired surface and the exact size. In every case the specimens had to be kept perfectly straight, and the taper at each end was machined on a lathe, holes were drilled, and the copper plugs were fitted. The copper plugs, once put in, were never removed ; when repeated tempering on certain cold-drawn specimens was carried out, the copper plugs were also tempered as if they belonged to the specimen. Tempering or annealing above 200° C. was conducted in a vacuum furnace, and below 200° C. in an oil bath, for which the temperature was easier to control. The specimens were thus kept free from oxidation.

*Procedure*—The specimens, rigidly mounted in the specimen holder, were immersed in the oil bath, 24 hr. being allowed for thermal equilibrium to be reached before measurements were taken. The battery, being fully charged, was allowed to settle down for at least 12 hr. before use, and a steady supply was usually reached about 5 min. after switch-on.

Readings were taken according to the following procedure (*see* Fig. 35) :

(*A*) With the reversing switch *M* in position *a* :

(i) Balance the voltage drop of $S_1$ on potentiometer *V* and that of the standard resistance *C* on *I* simultaneously

(ii) Switch over to $S_2$ and balance quickly without altering the setting on *I*

(iii) If the galvanometer spot (*z*) corresponding to potentiometer *I* has not moved at all, the readings for $S_1$, $S_2$, and the setting of *I* are taken down. If it has moved, but only slightly, the spot should be brought back to zero by adjusting *R* and the reading for $S_2$ subsequently taken

(iv) Operations (i) and (ii) are usually repeated about six times in each experiment, and the agreement between the corresponding readings must be within $\pm 0{\cdot}5$ μV.

(*B*) Change the switch *M* to position *b* and reverse the battery leads of *V* ; adjust the current to the same value and repeat the operations as in (*A*)

(*C*) Reverse *M* back to position *a* and check the previous results

(*D*) The difference in voltage drop between the two specimens ($S_1$ and $S_2$) does not remain the same when the current is reversed. This can only be due to thermal e.m.f. across the two specimens. In order to check and estimate the thermal e.m.f. the voltage drops across $S_1$ and $S_2$ are measured immediately after the current has been switched off, while the specimens are still hot. As explained later, this operation provides a check for the readings obtained in (*A*), (*B*), and (*C*).

**Calculation**

Let *R* and *R′* be the resistance of the known and unknown specimens $S_1$ and $S_2$, respectively, and *V* and *V′* the corresponding potential drops. Then, if the current employed is the same for $S_1$ and $S_2$ :

$$\frac{R' - R}{R} = \frac{V' - V}{V} \text{ or } \frac{\Delta R}{R} = \frac{\Delta V}{V} \quad \ldots\ldots\ldots\ldots(1)$$

*V* and *V′* are not given directly by the settings of the potentiometer because of the presence of the thermal e.m.f.'s. Let the thermal e.m.f.'s be *e* and *e′*, and $V_a$ and $V'_a$ the observed readings for $S_1$ and $S_2$, respectively, when the reversing switch *M* is in position *a*. Then

$$V_a = V + e \text{ and } V'_a = V' + e' \quad \ldots\ldots\ldots(2)$$

By changing the switch *M* to position *b*, the current is reversed, and if the intensity does not change and the thermal e.m.f.'s remain the same, the new potential drops, $-V$ and $-V'$, and the corresponding readings, $-V_b$ and $-V'_b$, will have the same relation as before. Thus :

$$-V_b = -V + e \text{ and } -V'_b = -V' + e' \quad \ldots(3)$$

The negative signs denote that the readings are obtained with the battery leads to the potentiometer inverted.

By combining equations (2) and (3), we have

$$\Delta V = V' - V = \tfrac{1}{2}[(V'_a - V_a) + (V'_b - V_b)]$$

Considering the small value of *e* and *e′*, the following relation may then be written :

$$\frac{\Delta R}{R} = \frac{\Delta V}{V} = \tfrac{1}{2}\left[\frac{V'_a - V_a}{V_a} + \frac{V'_b - V_b}{V_b}\right] \quad \ldots\ldots(4)$$

It can be seen that the results are only dependent on the ratios $V'_a/V_a$ and $V'_b/V_b$. Provided that each pair of readings is taken quickly enough, slight fluctuations of current before and after reversing and gradual changes in potentiometer calibration will not affect the results. Actually, several readings are taken before and after reversing and the average values of $V_a$, $V_b$, $V'_a - V_a$, and $V'_b - V_b$ are used in the calculation, according to the following equation :

$$\frac{\Delta R}{R} = \tfrac{1}{2}\left[\frac{V'_a - V_a}{V_a} + \frac{V'_b - V_b}{V_b}\right] \quad \ldots\ldots\ldots(4a)$$

The above calculation assumes that the thermal e.m.f.'s *e* and *e′* do not vary. In practice, if the temperature of the specimens increases, the thermal e.m.f.'s may gradually change. Even when the readings obtained in operation (*C*) agree with those in (*A*), it does not necessarily mean that the thermal e.m.f.'s remain constant, because they may vary with the direction of the current (Peltier effect or non-linear resistances at the current terminals, for instance). Operation (*D*) has therefore been designed as an additional check. By measuring the voltages when the current is off and the specimens are still hot, it gives a rough estimate of $e' - e$, to be compared with that given by

$$e' - e = \tfrac{1}{2}[(V'_a - V_a) - (V'_b - V_b)]$$

as can be derived from equations (2) and (3).

**Resistivity**

The value of $R' = R + \Delta R$, calculated from equation (4*a*), represents the resistance of the measured portion of the unknown specimen. To convert this into resistivity, several considerations have to be made. For instance, the resistance of a specimen may vary along its length, *i.e.*, it may depend upon where the knife-edges are placed. Since the distance between the knife-edges is fixed, the variation in resistance could be due only to the non-uniformity of the section or to the inhomogeneity of its properties.

Not only will a machined specimen show a variation in diameter along its length, but the fact that the entire length of a cold-drawn specimen passes through the same die does not necessarily ensure a uniform section. Thus, with high reductions, the diameter may gradually decrease from one end to the other, due, most probably, to the effect of heat generated during drawing. When several specimens of different reductions are finished through the same die, specimens of higher reductions tend to have a smaller average diameter. These differences are, of course, very small, but for a $\frac{1}{4}$-in. size a difference of 0·001 mm. in dia. means an error of 0·03% in the resistivity. In the present experiments the diameters of the specimens were measured at 22° C. at regular intervals along their length by a special Mercer gauge, and the mean diameters were worked out by integration.

In certain cases the section of a specimen may be extremely uniform, but the resistance along its length may not remain constant. This is associated with inhomogeneity of properties and can be due to many different causes, for instance, a temperature gradient existing during tempering, or a variable amount of reduction along the length resulting from an irregular diameter before drawing.

In the present experiments the variation in resistance due to these causes did not usually exceed 0·1%. With cold-drawn specimens of different reductions several portions of the specimen were measured along its length and the average resistivity

was given. For the study of change in resistivity on tempering, the same portion of the specimen was used throughout, and the reproducibility of the test was much higher, about 0·01%.

The calculation of the resistivity is based upon the following equation :

$$\frac{\Delta\rho}{\rho} = \frac{\Delta R}{R} - \left(\frac{\Delta l}{l} - \frac{\Delta A}{A}\right)\left[1 - \left(\frac{\Delta l}{l} - \frac{\Delta R}{R}\right)\right] \quad \ldots(5)$$

where $\Delta\rho = \rho' - \rho$. $\Delta R = R' - R$. $\Delta l = l' - l$. and $\Delta A = A' - A$

$\rho, \rho'$ = resistivity

$l, l'$ = effective length (*i.e.*, distance between knife-edges)

$A, A'$ = average cross-sectional area

$\rho$, $l$, and $A$ apply to specimen $S_1$, and $\rho'$, $l'$, and $A'$ apply to specimen $S_2$.

The second-order term in equation (5) is necessary only when the difference in section between the two specimens is appreciable. The correction $\Delta l/l$ can be obtained very easily by exchanging $S_1$ and $S_2$ in the specimen holder, and the average value of $\Delta R/R$ is taken for the calculation. Since this correction was small, it can be shown that equation (5) can be reduced to the following form :

$$\frac{\Delta\rho}{\rho} = \frac{\Delta R}{R} - \frac{\Delta l}{l} - \frac{2\overline{\Delta d}}{d} + \frac{\overline{\Delta d}}{d}\left(\frac{\overline{\Delta d}}{d} - \frac{2\Delta R}{R}\right) \quad \ldots(6)$$

in which $\overline{\Delta d}/\overline{d}$ is given by the mean diameters of the two specimens.

### Experimental Accuracy

With these arrangements and the various precautions, the readings in a given measurement could be repeated to within a few thousandths of a percent. This does not, of course, represent the overall experimental accuracy, and the main errors arise in the measurement of the dimensions of the specimen. For example, when a specimen was removed and replaced, the results were identical only within about 0·02%, largely because it was not possible to locate exactly the same measured portion. When different specimens are compared, the measurement of diameters may introduce an error of 0·03%. It may be concluded that the overall experimental accuracy was 0·05% when diameter measurements were involved, and only 0·02% when diameter measurements were unnecessary, as in the tempering experiments.

## RESULTS

### As-Drawn Specimens

The results for steel *D*1, showing the change of resistivity with respect to drawing reductions, are shown in Fig. 38*a* (full line). Before cold-drawing, the steel was lead-patented at 480° C. to give an initial structure of very fine pearlite. This treatment yielded the best drawing properties, and relatively high reductions could be obtained with large specimens. In Fig. 38*a* the resistivity of *D*1 decreases with increasing cold-drawing and, at about 70% reduction, the decrease is as much as 5½%. Tempering at 200° C. produced very little change in the resistivity of these cold-drawn specimens, as shown by the broken line (Fig. 38*a*) ; this feature agrees with the tempering results given later.

In another series of experiments, steel *D*1 was cold-drawn in the normalized state. The initial structure was lamellar, but somewhat coarser than that obtained by lead-patenting, and drawing was possible only to about 60% reduction. The results are given in Fig. 38*b*. Here, again, the resistivity decreased continuously with increasing drawing reductions. Thus, at about 60% reduction, the resistivity was 4% lower than that of the unworked specimen. This decrease was 1% less than that on lead-patented specimens for the corresponding reduction. In fact, by comparing Fig. 38*b* with Fig. 38*a*, it is seen that below about 20% reduction, the decrease in resistivity was rather similar in the two cases, but at higher reductions the decrease was definitely greater for the finer lamellar structure.

With the ingot iron *A*1, the resistivity increased on cold-drawing, Fig. 38*c*. Up to 60% reduction the resistivity varied with the degree of drawing almost linearly, and the amount of increase was relatively small, only about 0·8%. Beyond 60% reduction the increase was more rapid and the next 20% reduction caused an additional increase of 1%, making the total increase in resistivity about 1·8% at 80% reduction.

The fact that cold-work increases the resistivity of a low-carbon iron, but decreases that of a high-carbon steel, requires careful consideration. Clearly, the main difference between these two is the presence of carbon in the latter, but it could not have been a solution effect because the entering of carbon into solution gives rise to an increase, rather than a decrease, in resistivity. On the other hand, cementite has a higher resistivity than ferrite, and the alignment of the cementite along the drawing direction, as given by a fibre structure, might explain the results. In this case, the shape and size of the cementite as round particles or as lamellae, for example, would be expected to have a predetermined influence upon the electrical resistivity of steel after drawing. Accord-

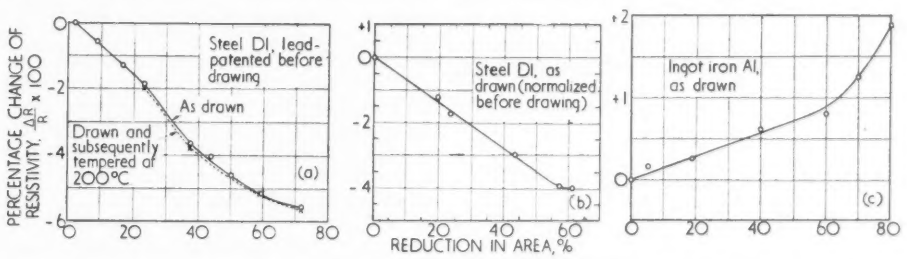

PERCENTAGE CHANGE OF RESISTIVITY, $\frac{\Delta R}{R} \times 100$
Steel DI, lead-patented before drawing
As drawn
Drawn and subsequently tempered at 200°C
(a)
Steel DI, as drawn (normalized before drawing)
(b)
Ingot iron AI, as drawn
(c)
REDUCTION IN AREA, %

**Fig. 38—Effect of cold-drawing upon resistivity of (*a*) steel *D*1, drawn from lead-patented condition, (*b*) steel *D*1, drawn from normalized condition, and (*c*) ingot iron *A*1**

ingly, further experiments were carried out on the same steel (*D*1), which was initially treated to a sorbitic condition so that the new results could be directly compared with those already reported for lamellar structures.

Hot-rolled bars, $1\frac{1}{8}$ in. dia., of *D*1 were soaked and water-quenched from 850° C., and were subsequently tempered at 650° C. for 3 hr. to give a sorbitic structure in which the cementite was spheroidized as round particles. Owing to the large size of bar, the quenching was not as effective in the centre as at the outside of the specimens, with the result that there were signs of incomplete spheroidization after tempering. However, as a whole, the structure was sorbitic, and the shape and size of the cementite particles were uniform in the centre, which was the portion concerned in the experiments. After heat-treatment, the bars were machined to various sizes and were subsequently drawn to $\frac{1}{4}$ in. dia. to give different reductions. The results are shown in Fig. 39.

Figure 39 shows that, with sorbitic specimens, the decrease in resistivity produced by cold-drawing was considerably less. At 70% reduction, the decrease was only 1·4%, as compared with $5\frac{1}{2}$% when the steel was drawn from a lamellar pearlite given by patenting. The 80% reduction specimen showed a higher resistivity than the 70% reduction, the difference being about 0·9%. A similar increase was observed when lamellar pearlite of the same steel was drawn more than 80% reduction. It has been concluded that the later increase in resistivity at very high reductions was due to internal flaws produced by overdrawing.

These results justify the contention that the shape, size, and alignment of the cementite play an important part in determining the electrical resistivity of steel. The limitation and effect of these factors will be dealt with by a theoretical treatment later in the paper.

### Change of Resistivity in Cold-Drawn Specimens after Tempering

A specimen of *D*1 (0·58% C), cold-drawn in the normalized condition to about 60% reduction, was tempered *in vacuo* at different temperatures for 1 hr., and the resistivity was measured after each tempering. The results are shown in Fig. 40*a*. The resistivity of the cold-drawn specimen decreased very slightly (0·05%) after tempering at 150° C., but increased on subsequent tempering to regain its original value at 200° C. Above 250° C., successive heating at higher temperatures decreased the resistivity, although the decrease was very slow below 350° C. From 350° C. upwards, the resistivity dropped rapidly with increasing tempering temperature, until a minimum was reached at 650° C. This minimum was 6% below the resistivity of the specimen in the as-drawn condition and 10% less than that of the unworked

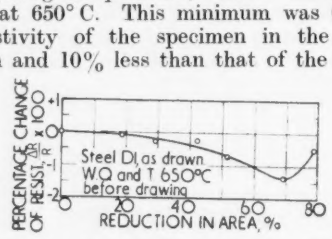



**Fig. 39—Effect of cold-drawing upon resistivity of steel *D*1, cold-drawn from the quenched and tempered condition**

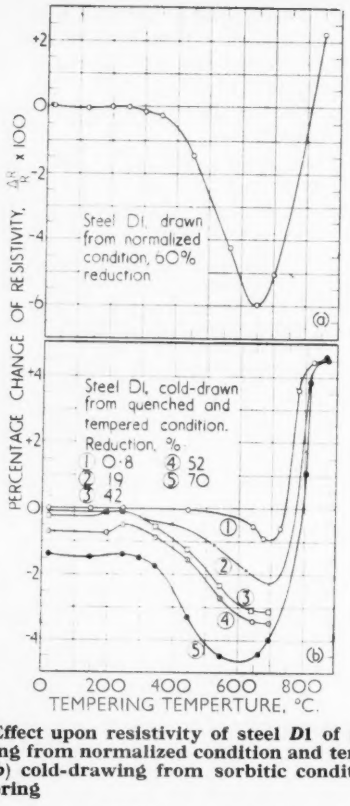



**Fig. 40—Effect upon resistivity of steel *D*1 of (*a*) cold-drawing from normalized condition and tempering and (*b*) cold-drawing from sorbitic condition and tempering**

specimen. Above 650° C. the resistivity increased very sharply. As pointed out before, this sharp increase is due mainly to a change of microstructure in which the fibre distribution of cementite is gradually broken up and finally becomes random pearlite as the tempering temperature is raised above the $Ac_1$ change. In general, the curve in Fig. 40*a* is similar to curve (2) in Fig. 33*b* for the same steel, but drawn in the annealed condition to 73·5% reduction. In the latter case, also, the specimen was much smaller and a different method of measurement was used.

The results of similar experiments on sorbitic specimens of various reductions are given in Fig. 40*b*. For reasons already stated, the 0·8% reduction specimen (curve (1)) was intended to represent the steel in the unworked condition, and even in this case the resistivity began to decrease when the tempering temperature was above 450° C.; a minimum was reached at 700° C., and this was 1% lower than the resistivity in the untempered condition.

As the steel was quenched and tempered (650° C. for 3 hr.) and the amount of cold-working given to the specimen was extremely small, being only enough to smooth-out its surface, the decrease in resistivity shown in curve (1) may be due mainly to the effect of prolonged tempering, rather than to the removal of the effect of cold-working. Indeed, it was found later that, with quenched specimens, tempering for at least 4 hr. at 650° C. was necessary for the resistivity to attain an approximately constant value.

This decrease must be considered with the results for the cold-worked specimens (curves (2)–(5)). These four curves have features in common and are similar to those observed with the cold-drawn pearlitic specimens, *viz.* : (1) The decrease in resistivity on tempering up to 200° C. was extremely small, not more than 0·1% ; (2) there was an increase in resistivity at 250° C.—the maximum effect was obtained with the 52% reduction specimen for which the increase was about 0·2% ; (3) in all cases the decrease in resistivity was rapid above 300–350° C., until a minimum was reached at 600–700° C. ; and (4) above the temperature of minimum resistivity the curves, including that of the unworked specimen, show a sharp rise and meet at about 870° C.

With regard to features (1) and (3), the decrease of resistivity in cold-worked specimens on tempering is to be expected on the grounds of the release of internal strains, slow at first but becoming increasingly fast as the temperature is raised, until such strains are removed completely. When the temperature is sufficiently high the shape and distribution of the cementite present will become affected. The minimum resistivity for each specimen is a resultant of at least two main factors, *viz.*, a maximum removal of internal strains, and a minimum change of the fibre distribution of cementite in the drawing direction. Figure 40*b* shows that the temperature at which the minimum resistivity occurs was lowered as the degree of cold-drawing of the specimen was increased, for example, 700° C. for 19% reduction, as compared with 600° C. for 70% reduction. Moreover, the value of the minimum resistivity was different for each specimen ; the higher the reduction, the lower was this value. Thus, the minimum resistivity of the 19% reduction specimen was only 1·3% lower than that of the 'unworked' one, but the corresponding difference between the 70% reduction specimen and the latter was 4%. Feature (4) was clearly due to a change of microstructure. The fibre arrangement of cementite particles which gives the least resistivity became gradually broken up and finally changed into random pearlite when annealing above the $Ac_1$ temperature. The curves (Fig. 40*b*) meet at 870° C., merely indicating that after annealing at that temperature the structure of the specimens became identical.

Feature (2) has also been observed with specimens cold-drawn from a pearlitic structure. As pointed out in a previous part, it is believed that certain changes take place in cold-worked cementite on heating and are directly responsible for the increase in resistivity described above. The fact that on tempering at 250° C. the increase in resistivity was slightly greater for the 52% reduction specimen than for the 70% reduction, does not necessarily mean that in the latter the cementite is less affected during cold-working. Bearing in mind the shape of the curves for cold-worked ingot iron in Fig. 33*a*, it is clear that the increase of resistivity in cold-worked high-carbon steels, in this as also in other cases, actually covered a wide range and was not confined to 250° C. To assess the total effect, the increase over the whole range of temperatures, which may spread from room temperature to 400–450° C., must be taken into account.

### Effect of Grain Size upon Resistivity in Iron

A number of ingot-iron specimens (*A*1, 0·04% C) were cold-drawn to different reductions and subcritically annealed to obtain different grain sizes. They were then machined to $\frac{1}{4}$ in. dia., and resistivity measurements were carried out in the usual manner. The treatment and the results obtained are summarized below :

*Grain-Size Treatment and Resistivity Results (Ingot Iron)*

| Specimen | Reduction, % | Annealed 1 hr. at : | Grain Size, grains in. | Resistivity, microhm-cm. | Change in Resistivity, % |
|---|---|---|---|---|---|
| *A* | 25·4 | 650° C. | 740 | 12·548 | 0 |
| *B* | 41·5 | .. | 1610 | 12·581 | 0·26 |
| *C* | 60·8 | .. | 2590 | 12·630 | 0·65 |
| *D* | 81·4 | .. | 3990 | 12·680 | 1·05 |
| *E* | 88·4 | 600° C. | 4520 | 12·652 | 0·83 |

Specimens *A*, *B*, *C*, and *D* were annealed at the same temperature for the same length of time, whereas the temperature for *E* was 50° C. lower. Figure 41 shows the relation between the initial drawing reduction and the number of grains produced after annealing at 650° C. for 1 hr. The points lie on a smooth curve and a large circle is drawn around each point to show the standard deviation obtained from frequency curves based on grain-size counts. When the grain size is plotted against the resistivity of the specimen, Fig. 42, it is seen that the resistivity increases with the decrease of grain size. Using specimen *A* as a standard, the resistivity was increased by 1·05% when the grain size of the specimen changed from 740 grains linear in. to 3990 grains linear in. The only exception was specimen *E* ; in spite of its smaller grain size, the resistivity of *E* was smaller than that of *D*, but specimen *E* was annealed at 600° C. instead of 650° C.

Bearing in mind the results given in Fig. 33*a*, it is hardly surprising that a cold-worked ingot-iron

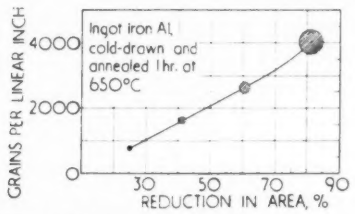



**Fig. 41—Effect of annealing at 650° C. upon grain size of ingot iron *Al***

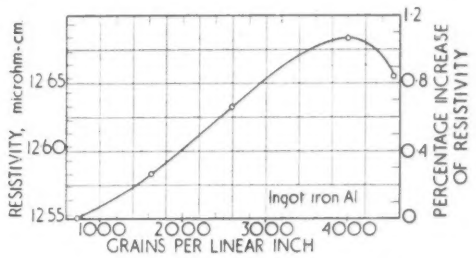



**Fig. 42—Effect of grain size upon resistivity of ingot iron *Al***

specimen annealed at 600° C. should have a lower resistivity than that annealed at 650° C. These results show that factors other than the grain size appear to affect the resistivity of subcritically annealed ingot-iron specimens. To assess the effect of grain size, the temperature and time of annealing should be kept constant.

## EFFECT OF CEMENTITE DISTRIBUTION UPON RESISTIVITY: THEORETICAL CONSIDERATION

### Lamellar and Fibre Structures

Only the ideal cases of these two structures can be dealt with. The first is concerned with an ideal fibre structure in which the cementite is present as continuous streaks parallel to the direction of the current. The conductivity is given by :

$$\lambda_m = (1 - v)\lambda + v\lambda' \quad \ldots\ldots\ldots\ldots\ldots\ldots(1)$$

where $\lambda_m = \frac{1}{\rho_m}$, $\lambda = \frac{1}{\rho}$, $\lambda' = \frac{1}{\rho'}$;

$\rho_m$, $\rho$, and $\rho'$ are the resistivity of the specimen, ferrite, and cementite, respectively, and $100v$ is the volume percentage of cementite. In the other case, it is an ideal lamellar structure in which alternate layers of ferrite and cementite are arranged across the whole section of the specimen, and the resistivity of such a structure will be :

$$\rho_m = (1 - v)\rho + v\rho' \quad \ldots\ldots\ldots\ldots\ldots\ldots(2)$$

For the present calculation, the values $\rho = 10$ microhm-cm. and $\rho' = 45$ microhm-cm. will be used throughout, but the validity of the equations is independent of the numerical values adopted. Substituting these values in equations (1) and (2), we have :

$$\lambda_m = \lambda(1 - 0{\cdot}777v) \quad \ldots\ldots\ldots\ldots\ldots (1a)$$

for the ideal fibre structure, and

$$\lambda_m = \lambda \frac{1}{1 + 3{\cdot}50v} \quad \ldots\ldots\ldots\ldots\ldots(2a)$$

for the ideal lamellar structure. It can be seen from these equations that with a 0·58% C steel, for which $v = 0{\cdot}090$, the difference in resistivity between the two structures is of the order of 25%.

The obvious conclusion which can be drawn from this calculation is that, with an ideal lamellar structure, a decrease in resistivity of up to 25% in a 0·58% C steel does not necessarily mean a change of constitution, because it can be explained on the basis of a change in cementite distribution. In practice, however, the pearlitic structure which is obtainable is very different from the ideal lamellar case assumed above because the lamellae are never continuous *across* the whole cross-section to offer maximum resistance to the flow of current.

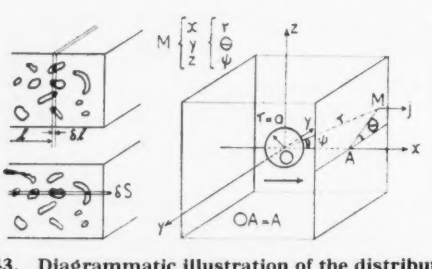



**Fig. 43. Diagrammatic illustration of the distribution of carbide particles in sorbite, with notations adopted for the calculation of electrical resistivity**

For a given steel the resistivity may vary with the treatment, such as annealing, normalizing, or patenting, although all these treatments give rise to a kind of pearlitic structure. A general survey of the results indicates that with a 0·58% C steel the resistivity of a pearlitic structure is about 10% higher than that of a sorbitic one, whilst such a difference between the latter and an ideal lamellar structure should be about 20%, as will be clear later.

### Spheroidized Structure or Sorbite

Sorbite consists of small particles of cementite, approximately spherical in shape, and randomly distributed throughout. In an attempt to calculate and show the effect of change in cementite distribution upon the resistivity of such a structure, we may start with an ideal case in which all the cementite particles are assumed to be spheres arranged in a cubic order, *i.e.*, each sphere (radius $a$) occupies the centre of a cubic cell of edge $2A$. This is illustrated in Fig. 43, together with the notations adopted in the calculation. The principle of calculation consists of working out the current flowing under a given potential difference. The details involved in the calculation are somewhat lengthy, and only an outline of the important steps is given here.

The first step is to determine the density of current $J$, at each point. This current density is linked up with the potential $-V$, by the relation :

$$\vec{J} = \lambda \overrightarrow{\text{grad}}\ V$$

in which $\overrightarrow{\text{grad}}\ V$ is a vector of the components, $\partial V/\partial x$, $\partial V/\partial y$, $\partial V/\partial z$, and $\lambda$ is the conductivity at the potential point. The function $V$ is known to be a solution of :

$$\frac{\partial^2 V}{\partial x^2} + \frac{\partial^2 V}{\partial y^2} + \frac{\partial^2 V}{\partial z^2} = 0$$

subjecting to certain boundary conditions. The boundary conditions are (1) that the three planes, $x = 0$, and $x = \pm A$, should be equi-potential, *i.e.*, $x = 0$, $V = 0$ ; $x = \pm A$, $V = \pm V_A$, and (2) the expressions which will satisfy the continuity of the current from outside to inside the sphere. The latter give rise to :

$$\lambda \frac{\partial V}{\partial r} = \lambda' \frac{\partial V'}{\partial r}$$

with $V = V'$ on the surface of the sphere ($r = a$), $V$ and $V'$ being the potentials outside and inside the sphere, respectively.

It is convenient to tackle the problem by means of spherical co-ordinates. The general expression is

$$V = \sum_{1}^{\infty} \left(A_n r^n + \frac{B_n}{r^{n+1}}\right) S_n$$

$$= \left(A_1 r + \frac{B_1}{r^2}\right) S_1 + \ldots \left(A_n r^n + \frac{B_n}{r^{n+1}}\right) S_n + \ldots$$

in which $S_n$ are spherical harmonics (zonal to the approximation required), and $A_n$ and $B_n$ are numerical coefficients. In this expression, it has only been possible to find the first term, which is :

$$V = A_1\left(r + \frac{na^3}{r^2}\right)\cos\psi, \text{ where } n = \frac{\lambda - \lambda'}{2\lambda + \lambda'}$$

The current $C$ $\left(C = \lambda \iint \frac{\partial V}{\partial x}\, dS\right)$ flowing through the face of the cube ($x = A$) is :

$$C = 4A_1A^2\lambda\left(1 - \frac{na^3}{A^3}\frac{\sqrt{3}}{3}\right)$$

From this, the conductivity can be expressed as :

$$\lambda_m = \lambda\frac{1 - qv}{1 + pv} \quad \ldots\ldots\ldots\ldots\ldots\ldots(3)$$

where $p = \frac{6}{\pi}n$ and $q = p\frac{\sqrt{3}}{3}$

When $v$ is not too large, equation (3) can be written :

$$\lambda_m = \lambda\,[1 - (p + q)v + p(p + q)v^2]$$

Substituting $n$ for $p$ and $q$, we have :

$$\lambda_m = \lambda(1 - 3{\cdot}01nv + 5{\cdot}75n^2v^2) \quad \ldots\ldots\ldots\ldots(3a)$$

In the case of steel (cementite and ferrite), $n = 0{\cdot}35$, therefore :

$$\lambda_m = \lambda\,(1 - 1{\cdot}05v + 0{\cdot}70v^2) \quad \ldots\ldots\ldots\ldots(3b)$$

Having completed this calculation, the authors came across a paper by Lord Rayleigh,[27] dealing with the calculation of the dielectric constant of a medium containing spheres arranged in a rectangular order. His method of calculation was somewhat similar to that of the authors, but the main difference is that Lord Rayleigh used the Green theorem to evaluate the current. The relation was given by Lord Rayleigh as

$$\frac{\lambda_m}{\lambda} = 1 - \frac{4\pi B_1}{4A^2V_1}$$

In this equation $V_1$ is the periodic potential drop, but this is true only when the integration—the determination of $V$—is carried out far enough so that the faces of the cube are practically equi-potential. In view of this, the determination of the ratio $B_1/V_1$ is extremely complicated. However, in Lord Rayleigh's paper[27] this had been carried out. It is possible, therefore, to compare Lord Rayleigh's final equation with the authors'. Neglecting the high-power terms (greater than $v^4$), Lord Rayleigh's final equation can be written in the present notation as :

$$\frac{\lambda_m}{\lambda} = 1 - \frac{3nv}{1 + nv} \quad \ldots\ldots\ldots\ldots\ldots\ldots(4)$$

This equation can be reduced to the form :

$$\lambda_m = \lambda(1 - 3nv + 3n^2v^2) \quad \ldots\ldots\ldots\ldots(4a)$$

within the accuracy of 1/10,000.

Comparing the authors' equation ($3a$) with Lord Rayleigh's ($4a$), the only difference is the second-power term ; this difference amounts to $2{\cdot}75n^2v^2$.

Substituting the value of $n$ for steel in equation ($4a$) :

$$\lambda_m = \lambda(1 - 1{\cdot}05v + 0{\cdot}37v^2) \quad \ldots\ldots\ldots\ldots(4b)$$

It can be shown that for a 0·58% C steel ($v \simeq 0{\cdot}1$), the error introduced by the authors' equation ($3b$) instead of Lord Rayleigh's ($4b$) is not more than 0·3% in terms of the resistivity of the specimen ($\lambda_m$). It should be pointed out, however, that when $v$ is large, Lord Rayleigh's equation gives a better approximation than the authors'.

Bruggeman[28] has tried to apply Lord Rayleigh's equation to the case of a medium containing spheres of mixed sizes, and gave a formula which can be used for the calculation of conductivity. Bruggeman's formula is :

$$1 - v = \frac{\lambda' - \lambda_m}{\lambda' - \lambda}\sqrt[3]{\frac{\lambda}{\lambda_m}} \quad \ldots\ldots\ldots\ldots(5)$$

in which $\lambda_m$ appears as an implicit function of $v$ and cannot be put into the form of $\lambda_m = f(v)$.

Bruggeman's formula has been tested experimentally by a French worker,[29] who used microscopical dielectric spheres randomly distributed in another dielectric medium. The experimental results were in excellent agreement with Bruggeman's formula for all values of $\lambda/\lambda'$ up to infinity, as well as for different values of $v$, including the extreme case where the spheres are in contact with one another.

It is desirable to compare Bruggeman's formula with Lord Rayleigh's equation and the authors' in the case of a 0·58% C steel, for which $\lambda/\lambda'$ is 4·5 and $v$ is approximately 0·1. Although $\lambda_m$ in Bruggeman's formula cannot be put explicitly as a function of $v$, it is, however, possible to develop it into this form by means of MacLaurin's theorem. To do this, the successive derivatives are worked out by means of partial derivation of the two members in equation (5). Neglecting $v^3$ and higher-power terms, we have :

$$\lambda_m = \lambda\left[1 + 3\frac{\lambda' - \lambda}{\lambda' - 2\lambda}v + 3\frac{(\lambda' - \lambda)^2(\lambda + 2\lambda')}{(\lambda' + 2\lambda)^3}v^2\right]$$

This can be written :

$$\lambda_m = \lambda\left[1 - 3nv + 3n^2v^2\frac{\lambda + 2\lambda'}{\lambda' + 2\lambda}\right] \quad \ldots\ldots\ldots(6)$$

Equation (6) is the same as Lord Rayleigh's except that in this case the second-power term contains a multiplying factor of $(\lambda + 2\lambda')/(\lambda' + 2\lambda)$. For steel, the value of this factor is 0·65. Substituting in equation (6), we have :

$$\lambda_m = \lambda(1 - 3nv + 1{\cdot}95n^2v^2) \quad \ldots\ldots\ldots\ldots(6a)$$

and

$$\lambda_m = \lambda(1 - 1{\cdot}05v + 0{\cdot}24v^2) \quad \ldots\ldots\ldots\ldots(6b)$$

For $v = 0{\cdot}1$, the difference between equation ($6b$) and Lord Rayleigh's equation ($4b$) is only 0·1% in terms of the resistivity. If we accept the experimental proof[29] of Bruggeman's formula, the same proof should hold good for Lord Rayleigh's equation and for equation (3) for steel. With a 0·58% C steel, both Lord Rayleigh's equation and the authors' are valid in a practical case to within 0·5% when they are applied to calculate the resistivity in specimens containing spherical cementite particles of various sizes in random distribution.

### Practical Consideration of the Effect of Cementite Alignment in Spheroidized Sorbite

Using Lord Rayleigh's formula ($4b$), together with equation ($1a$), it can readily be seen that a change of structure from an ideal spheroidized to an ideal fibre arrangement will bring about an increase of conductivity, given by :

$$\frac{\Delta\lambda}{\lambda} = 0{\cdot}273v - 0{\cdot}37v^2$$

For a 0·58% C steel, for which $v \simeq 0{\cdot}1$, this increase is only 2·4%.

The limits between 0% and 2·4% may be much too wide for practical purposes. In practice such as cold-drawing, the alignment of cementite particles differs in degrees according to the reduction given to the specimen. Even in the extreme case, the spherical shape of the particles is not destroyed and the limiting structure is that the particles touch one another to form rows of continuous 'chains' parallel to the direction of drawing. Clearly, this is not quite the same as the ideal fibre structure which has been postulated. For convenience, we shall refer to the various alignments of cementite particles in spheroidized sorbite as 'chain structures' ranging from particles separated by a regular, but unspecified, distance in a row, to a limiting case in which adjacent particles are in contact with one another to form a continuous chain along the length of the specimen.

In an attempt to calculate the conductivity of the various 'chain structures' described above, the following treatment has been adopted. Each chain or each row of particles is enclosed in an infinitely long prism which has a square cross-section. The edge of this square is denoted by $2A$, which also represents the spacing of the spherical particles. Let the ratio between the total volume of all such prisms and that of the specimen be $\alpha$, and the cementite concentration inside the prism be $\omega$. By definition, $\omega = v/\alpha$, where $100v$ is the volume percentage of the cementite present. The value of $\omega$ is usually large, and the approximation given in equation (3) will no longer be valid. Lord Rayleigh's formula, equation (4), appears to be more suitable, but as the 'chain structures' approach the limiting case, in which the spheres touch one another, *i.e.*, $\omega = \pi/6 = 0{\cdot}52$, even equation (4) is not sufficiently accurate for the purpose. A better approximation would be a formula valid for large values of $v$, and this was given by Lord Rayleigh[27] as :

$$\lambda_m = \lambda\left[1 - \frac{3nv}{1 + nv - \beta nv^{\frac{10}{3}}}\right] \quad \ldots\ldots\ldots\ldots(7)$$

in which $n = \dfrac{\lambda - \lambda'}{\lambda' + 2\lambda}$ as before, but $\beta = 1{\cdot}65\ \dfrac{\lambda - \lambda'}{\lambda' + \frac{3}{4}\lambda}$.

Without sacrificing any accuracy, equation (7) can be written in the form :

$$\lambda_s = \lambda\left(1 - 3n\omega + 3n^2\omega^2 - 3n^3\omega^3 + 3n^4\omega^4 - 3\beta n^2\omega^{\frac{13}{3}}\right) \ldots(7a)$$

where $\lambda_s$ is the conductivity of one of the prisms defined above.

Substituting the values of $\lambda$ and $\lambda'$, $\beta$ is about 0·83 and the coefficient of $\omega^{\frac{13}{3}}$ in equation (7*a*) is 0·30. In the case of a limiting 'chain structure' $\omega = \pi/6 = 0{\cdot}52$ and the last term in equation (7*a*), $3\beta n^2\omega^{\frac{13}{3}}$, will be equal to 0·017 or 1·7% of $\lambda$ and, therefore, appears to be important.

Treating the prisms as members of a separate constituent, the conductivity of a 'chain structure' can be written in a form identical to that for an ideal fibre structure, *i.e.* :

$$\lambda_m = (1 - \alpha)\lambda + \alpha\lambda_s$$

$\lambda_s$ being given by equation (7*a*). Therefore :

$$\frac{\lambda_m}{\lambda} = 1 - 3nv\left(1 - n\omega + n^2\omega^2 - n^3\omega^3 + \beta n\omega^{\frac{10}{3}}\right) \ldots(8)$$

To check the accuracy of equation (8), as applying to 'chain structures' in a 0·58% C steel, for which $v \simeq 0{\cdot}1$, it can be shown that, even for the limiting case where $\omega = \pi/6$, the last term contributes only 0·35% of the ratio $\lambda_m/\lambda$. The term $\omega^{\frac{13}{3}}$ was the highest available in Lord Rayleigh's formula ; had it been possible to develop still higher-power terms, such terms would presumably be less and less important. For practical purposes, equation (8) is a good approximation.

For the limiting 'chain structures' in spheroidized steel, $\omega = \pi/6$, and $n\omega = 0{\cdot}183$. Substituting these values in equation (8) :

$$\frac{\lambda_m}{\lambda} = 1 - 2{\cdot}63nv \quad \ldots\ldots\ldots\ldots\ldots\ldots(8a)$$

Comparing equation (8*a*) with equation (4*a*), it can readily be seen that the maximum effect of the alignment of cementite particles in a sorbitic steel upon its conductivity is given by :

$$0{\cdot}37nv - 3n^2v^2$$

For a 0·58% C steel ($v \simeq 0{\cdot}1$) this is equal to 0·9%.

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**The remaining Sections of this paper will be published in the August, 1950, issue of the *Journal*, namely : Section III—X-Ray Investigations of Internal Strains in Cold-Drawn Steels ; Section IV—Effect of High-Speed Deformation on Steel ; Section V—X-Ray Investigations of Structural Changes in Steel due to Cold-Working ; Section VI—Effect of Cold-Work on Hydrogen in Steel ; Section VII—General Discussion.**

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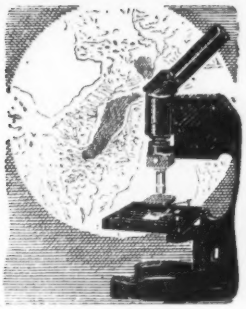

BRITISH IRON AND STEEL RESEARCH ASSOCIATION

# Gas-Pickling of Steel

*By J. Pearson, Ph.D., M.Sc., F.R.I.C., W. Bullough, B.Sc., A.R.I.C., and T. C. Canning*

**SYNOPSIS**

The treatment of steel strip in atmospheres containing gaseous hydrochloric acid has been practised in the U.S.A. for preparing the material for subsequent hot-galvanizing. Laboratory investigations have confirmed that galvanized coatings adhere more tightly to steel prepared in this way than to steel pickled in aqueous sulphuric acid. The attack on steel by gaseous hydrogen chloride differs from that by aqueous acids. The process is, however, suitable only for steel which can be heated to temperatures of about 750° C. without loss of required properties. Steel which must be finished in a cold-worked condition or with special heat-treatment cannot therefore be submitted to gas-pickling. Neither is the process suitable for the removal of thick scale ; the rapid attack of the gaseous HCl on any bare steel revealed by non-uniform removal of scale, leads to heavy metal losses and marked unevenness of surface.

GAS-PICKLING, in which steel is subjected to the high-temperature action of gas mixtures containing HCl vapour, is in use in the U.S.A. for treating steel strip before hot-galvanizing. It is claimed that the process produces surfaces to which metallic coatings adhere more tightly than to those pickled in aqueous media.* This claim has been investigated and gas-pickling in general examined, since it is a dry process which might offer an alternative to those pickling practices which produce embarrassing amounts of spent liquor.

The pickling atmospheres employed in America are obtained by burning mixtures of chlorine and hydrocarbon gas and diluting with flue gas, the final composition being approximately 70% $N_2$, 20% HCl, and 10% $CO_2$, by volume. The pickling temperature is about 730° C. (1350° F.). The aspects requiring study were :

(1) The influence of temperature, time of treatment, concentrations of HCl and $CO_2$, and rate of gas-flow, on loss in weight and surface roughness

(2) The nature of the attack on the steel surface, to determine whether it differed from that produced by aqueous pickling

(3) The efficiency with which high-temperature scale was removed

(4) The possibility of formation of CO during pickling

(5) The adhesion of subsequently applied hot-dipped coatings, *e.g.*, zinc.

## EXPERIMENTAL

### Pickling of Steel

Experiments were conducted in the temperature range 600–900° C., with gas compositions varying from 0–20% $CO_2$, 2·5–20% HCl, and with various gas-flow rates. The steel used was principally cold-reduced, inert-atmosphere-annealed, temper-passed strip, as made for tinning. It was rolled from rimmed ingots, and the composition of the rim, which was the only part attacked during the majority of experiments, was :

0·037% C, 0·020% S, 0·013% P, 0·11% Cu, 0·35% Mn, 0·09% Ni, 0·02% Cr, 0·007% Si.

Supplies of $N_2$ and $CO_2$, at rates determined by passing through separate flow-meters, were freed from

* R. F. Renkin : *Steel*, 1944, vol. 114, Mar. 27, pp. 102–104; J. J. Turin: *Iron Age*, 1944, vol. 153, Apr. 20, pp. 64–70.

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oxygen in chromous chloride wash-bottles and dried by passage through $H_2SO_4$ and over anhydrone (magnesium perchlorate). The HCl was generated by the action of concentrated $H_2SO_4$ on $NH_4Cl$ pellets and dried by passing through a sulphuric acid bubbler, the rate of flow being indicated by a sulphuric acid-filled flow-meter. The $N_2$, $CO_2$, HCl mixture was then passed through a furnace tube 2 ft. × 1 in. dia., the central portion of which was heated to the desired temperature. Initially the furnace tube was swept out with dry oxygen-free nitrogen and a weighed steel specimen (1 in. × $\frac{7}{8}$ in. × 30 gauge) was introduced, attached to the end of a long steel rod. When the pyrometer in the furnace tube again indicated that the temperature was reached, the ternary gas mixture was substituted for the $N_2$ for a given period and finally $N_2$ alone was again passed. The specimen was withdrawn to the end of the tube and allowed to cool in the $N_2$ stream. It was then detached from the steel rod, given a quick wash in ammonium tartrate solution, water, and methylated spirit, and finally dried, reweighed, and examined microscopically and on the Talysurf meter. Specimens were also pickled in 6% wt./vol. $H_2SO_4$ at 85° C. to give losses in weight similar to those obtained by gas-pickling.

Experiments were also conducted by replacing the steel strip with pieces of rimmed-steel rod, 3 in. × $\frac{1}{4}$ in. dia., which had been scaled at high temperature, to study the efficiency with which this scale was removed.

To investigate the proportion of CO formed, the apparatus was modified. The specimen was introduced into the furnace in a fireclay boat, so as to avoid attack on the steel rod. The gases issuing from the furnace after pickling were passed over KOH pellets and Carbest to remove HCl, $CO_2$, and $FeCl_2$, and over $CaCl_2$ to remove any water. The $H_2$ and CO formed during the reaction were then passed over heated palladinized asbestos in the presence of excess $O_2$, and the resulting water and $CO_2$ were collected in weighed tubes containing $CaCl_2$, and Carbest and $CaCl_2$, respectively.

### Adhesion of Hot-Galvanized Coatings

Specimens 5 in. × $\frac{7}{8}$ in. were pickled for $2\frac{1}{2}$ min. in 20% HCl, 80% $N_2$ at 700° C., and in 20% HCl, 10% $CO_2$, 70% $N_2$ at 650° C., to produce an attack on the steel similar to that shown in Fig. 6*a* (equivalent to 11·5 μ-in. removed). After cooling, the samples were washed in ammonium tartrate solution, water, and methylated spirit, dried, and pre-fluxed by warming to 60° C. and dipping in a solution of $ZnCl_2$/$NH_4Cl$. They were then galvanized by immersing vertically for 20 sec. in molten pure zinc at 440° C.

The galvanized strips were cut in half lengthways and given one acute bend, $\frac{1}{16}$ in. rad., in a former, the bend then being mounted in Bakelite and examined microscopically after polishing and etching.

Other specimens after galvanizing were given a thick electrodeposit of nickel, clamped in a vice, and the edges filed away. Where possible, a fine saw-cut was made at one end of the specimen to give purchase to two pairs of pliers used to tear the 'sandwich' apart. Samples which had been pickled in $H_2SO_4$ and galvanized were also given the same treatment, and, for interest, the adhesion tests were applied to some commercially galvanized sheets.

## RESULTS

### Pickling in Nitrogen and Hydrogen Chloride Only

*Loss in Weight and Surface Roughness*—Some typical results indicating the influence of temperature and time of treatment are given in Tables I and II, mean results at all temperatures being shown graphically in Figs. 1 and 2. The effects of concentration of HCl in the gas mixture and rate of flow of gas are shown in Fig. 3.

*Nature of Attack on Steel Surface*—Figure 6 shows two types of attack on the steel surface, one by gas pickling in a 20% HCl, 80% $N_2$ mixture, and the other by aqueous 6% $H_2SO_4$ at 85° C., for times such that the weight losses by both methods were of the same order. The nature of the attack by gaseous HCl under other conditions is essentially the same, being governed only by the amount of steel removed. The losses in weight and the equivalent thicknesses of metal removed are shown in Table III.

*Adhesion of Hot-Galvanized Coatings*—Photomicrographs of specimens which had been gas- or aqueous

### Table I

EFFECT OF TEMPERATURE

**Gas composition, 20% HCl, 80% $N_2$ ; rate of gas-flow, 400 ml./min. ≡ linear velocity of 3·2 ft./min. ; time of treatment, 5 min.**

| Temp., °C. | Loss in Weight, g./sq. in. | Thickness removed,* μ-in. | Average Roughness, μ-in. |
|---|---|---|---|
| 600 | 0·0006 | 4·7 | 7·5 |
| 650 | 0·0015 | 11·5 | 8 |
| 700 | 0·0041 | 32 | 10 |
| 750 | 0·0146 | 114 | 16 |
| 800 | 0·0343 | 268 | 41 |

* Thickness of steel removed from each face of the specimen

### Table II

EFFECT OF TIME OF TREATMENT

**Gas composition, 20% HCl, 80% $N_2$ ; rate of gas-flow, 400 ml./min. ; temperature, 800° C.**

| Time, min. | Loss in Weight, g./sq. in. | Thickness removed, μ-in. | Average Roughness, μ-in. |
|---|---|---|---|
| 1 | 0·0051 | 40 | 8 |
| $2\frac{1}{2}$ | 0·0141 | 110 | 22 |
| 5 | 0·0343 | 268 | 41 |
| 10 | 0·0850 | 665 | 74 |
| 20 | 0·1400 | 1095 | 99 |
| 25 | 0·1930 | 1509 | ... |
| 30 | 0·2870 | 2244 | ... |

### Table III

EFFECT OF NATURE OF TREATMENT

| Method | Loss in Weight, g./sq. in. | Thickness removed, μ-in. |
|---|---|---|
| **Gas-Pickling** | | |
| (*a*) | 0·0015 | 11·5 |
| (*b*) | 0·0041 | 32 |
| (*c*) | 0·0146 | 114 |
| (*d*) | 0·0343 | 268 |
| **Sulphuric Acid-Pickling** | | |
| (*f*) | 0·0014 | 10·7 |
| (*g*) | 0·0036 | 27·6 |
| (*h*) | 0·0121 | 94 |
| (*i*) | 0·0292 | 226 |

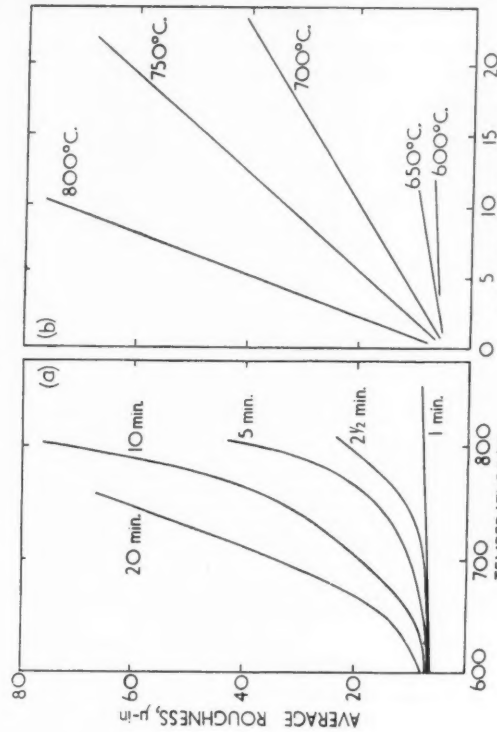


Fig. 1—Effect on loss in weight of (*a*) temperature, (*b*) time of treatment. Flow-rate, 400 ml./min.




Fig. 2—Effect on surface roughness of (*a*) temperature, (*b*) time of treatment. Flow-rate, 400 ml./min.




Fig. 3—Effect on loss in weight of (*a*) hydrogen chloride concentration, (*b*) flow-rate. Temperature, 800° C.; time of treatment, 5 min.

$H_2SO_4$-pickled before galvanizing, and of commercial galvanized sheets, after having been subjected to one bend, are depicted in Fig. 7. Galvanized strips plated with a thick nickel deposit and the edges subsequently filed away gave the following results :

*Gas-pickled, laboratory galvanized*—Good adhesion, no rupture on filing. On separation of the sandwich, the coating ruptured partly in the zinc and partly at the alloy/steel interface

*Sulphuric acid-pickled, laboratory galvanized*—Poor adhesion. Coating broke away from the steel base during filing

*Commercial Samples*—(1) Very poor adhesion, the coating breaking away at the alloy/steel interface during filing. (2) Good adhesion, no parting during filing. On separation of the sandwich, the coating ruptured mainly in the zinc and partly at the alloy/steel interface. This sample had been galvanized by the Sendzimir process.

### Pickling in Nitrogen, Hydrogen Chloride, Carbon Dioxide Mixtures

*Loss in Weight*—The influence of temperature and time of treatment on the loss in weight in mixtures containing $CO_2$ is shown in Tables IV and V and in Figs. 1*a* and 4. The influence of $CO_2$ concentration is given in Table VI and Fig. 5.

*Nature of Attack on Surface*—The type of attack on the steel surface was found to be identical with that in HCl, $N_2$ mixtures for corresponding losses in weight. The metal forming the rim or the core in the samples of rod treated for scale removal was found to show the same type of intergranular penetration as the strip steel.

*Efficiency of Scale Removal*—Since in industrial practice gas-pickling mixtures contain $CO_2$, descaling

## Table IV

### EFFECT OF TEMPERATURE

**Rate of gas-flow, 400 ml./min. ; time of treatment, 5 min.**

| Temp., °C. | Loss in Weight, g./sq. in. 10% $CO_2$ ; 20% HCl ; 70% $N_2$ Mixture | 20% HCl ; 80% $N_2$ Mixture |
|---|---|---|
| 600 | 0·0013 | 0·0006 |
| 650 | 0·0044 | 0·0015 |
| 700 | 0·0156 | 0·0041 |
| 750 | 0·0291 | 0·0146 |
| 800 | 0·0428 | 0·0343 |
| 850 | 0·0570 | 0·0685 |
| 900 | 0·0663 | 0·0815 |

## Table V

### EFFECT OF TIME OF TREATMENT

**Rate of gas-flow, 400 ml./min. ; temperature 800° C.**

| Time of Treatment, min. | Loss in Weight, g./sq. in. 10% $CO_2$ ; 20% HCl ; 70% $N_2$ Mixture | 20% HCl ; 80% $N_2$ Mixture |
|---|---|---|
| 5 | 0·0453 | 0·0343 |
| 10 | 0·1226 | 0·0850 |
| 15 | 0·1560 | ... |
| 20 | 0·2144 | 0·1400 |
| 25 | ... | 0·1930 |
| 30 | 0·3374 | 0·2870 |

experiments were conducted with such a mixture only. Using a $N_2$, $CO_2$, HCl (70 : 10 : 20) atmosphere at 800° C., qualitative tests showed that the $\frac{1}{2}$-in. portion of the 3-in. rod nearest the gas entry was descaled in half a minute. For total descaling of the rods the time required was $3\frac{1}{2}$–5 min., there being a decrease in concentration of HCl along the length of the rod due to rapid reaction with the steel. During this period the front portions of the rods were severely over-pickled. Non-uniform removal of scale thus leads to pronounced metal loss.

*Adhesion of Hot-Galvanized Coatings*—The results were essentially similar to those obtained with strips pickled in the absence of $CO_2$.

*Formation of Carbon Monoxide during Pickling*—The influence of temperature, time of treatment, and

## Table VI

### EFFECT OF CARBON DIOXIDE CONCENTRATION

| Temp., °C. | $CO_2$ Concentration, % | Loss in Weight, g./sq. in. |
|---|---|---|
| 800 | 0 | 0·0343 |
| 800 | 5 | 0·0429 |
| 800 | 10 | 0·0453 |
| 800 | 20 | 0·0463 |
| 650 | 0 | 0·0015 |
| 650 | 10 | 0·0044 |
| 650 | 20 | 0·0046 |

## Table VII

### FACTORS INFLUENCING THE FORMATION OF CARBON MONOXIDE

| Temp., °C. | Time of Treatment, min. | $CO_2$ in Mixture, % | CO in Exit Gases, % |
|---|---|---|---|
| 800 | 5 | 10 | 0·11 |
| 650 | 5 | 10 | 0·08 |
| 800 | 5 | 10 | 0·11 |
| 800 | 10 | 10 | 0·10 |
| 800 | 20 | 10 | 0·09 |
| 800 | 5 | 10 | 0·11 |
| 800 | 5 | 20 | 0·14 |

$CO_2$ concentration in the gas mixture on the formation of CO is shown in Table VII.

## DISCUSSION

### Loss in Weight and Surface Roughness

*Influence of Temperature*—As was stated above, roughness of a sample depended only on the loss in weight. In fact, when roughness was plotted against loss in weight, an essentially straight-line relationship was found, irrespective of the conditions under which pickling was done. Any remarks about loss in weight are therefore also true about change in roughness.

Tables I and IV and Figs. 1*a* and 2*a* show that the effect of temperature is normal, *i.e.*, an increase in temperature causes an increased rate of attack. Calculations show that the temperature coefficient is approximately 1·2/10° C. This implies that the reaction by which iron is removed from the surface, as $FeCl_2$, is controlled by a physical process. This is presumably the rate of diffusion of HCl molecules to the surface, the diffusion of reaction products ($FeCl_2$ and $H_2$) away from it, or a combination of the two. The rate of adsorption of HCl and of desorption of reaction products may also play a part in controlling the reaction rate.

*Effect of Time of Treatment*—Figures 1*b*, 2*b*, and 4 show that, at least during the earlier stages, loss in weight is directly proportional to treatment time. This is to be expected, but indicates that no residues are left on the surface to interfere with further progress of the reaction. In the absence of $CO_2$

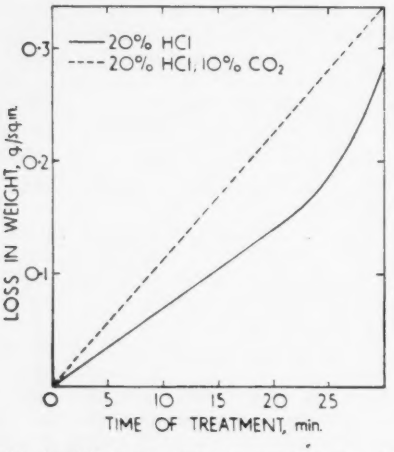


**Fig. 4—Effect on loss in weight of time of treatment. Flow-rate, 400 ml./min. ; temperature, 800° C.**

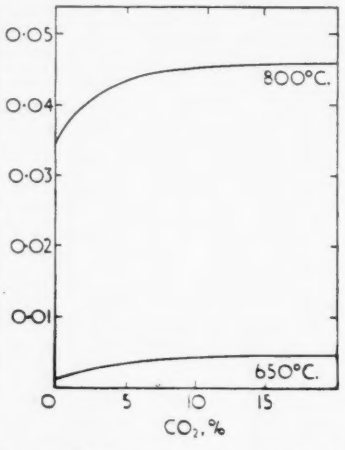


**Fig. 5—Effect on loss in weight of carbon dioxide concentration. Flow-rate, 400 ml. min. ; 20% hydrogen chloride ; time of treatment, 5 min.**

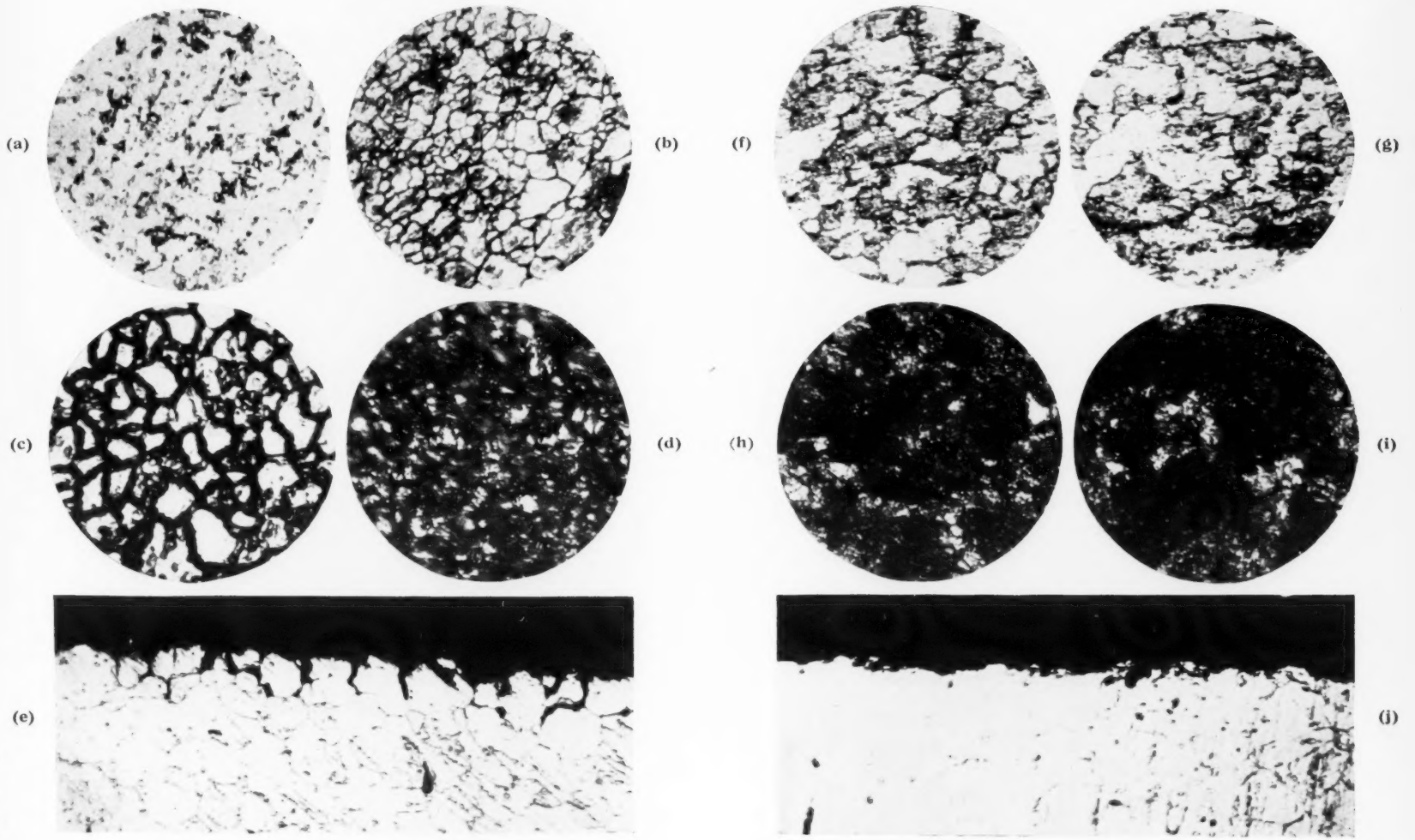


Fig. 6—Comparison of surface attack after various times of treatment, (*a-d*) by gas-pickling and (*f-i*) by sulphuric acid-pickling. Figs. (e) and (*j*) are cross-sections of (*c*) and (*h*) respectively × 500

[*Pearson, Bullough and Canning*
[*To face p.* 188

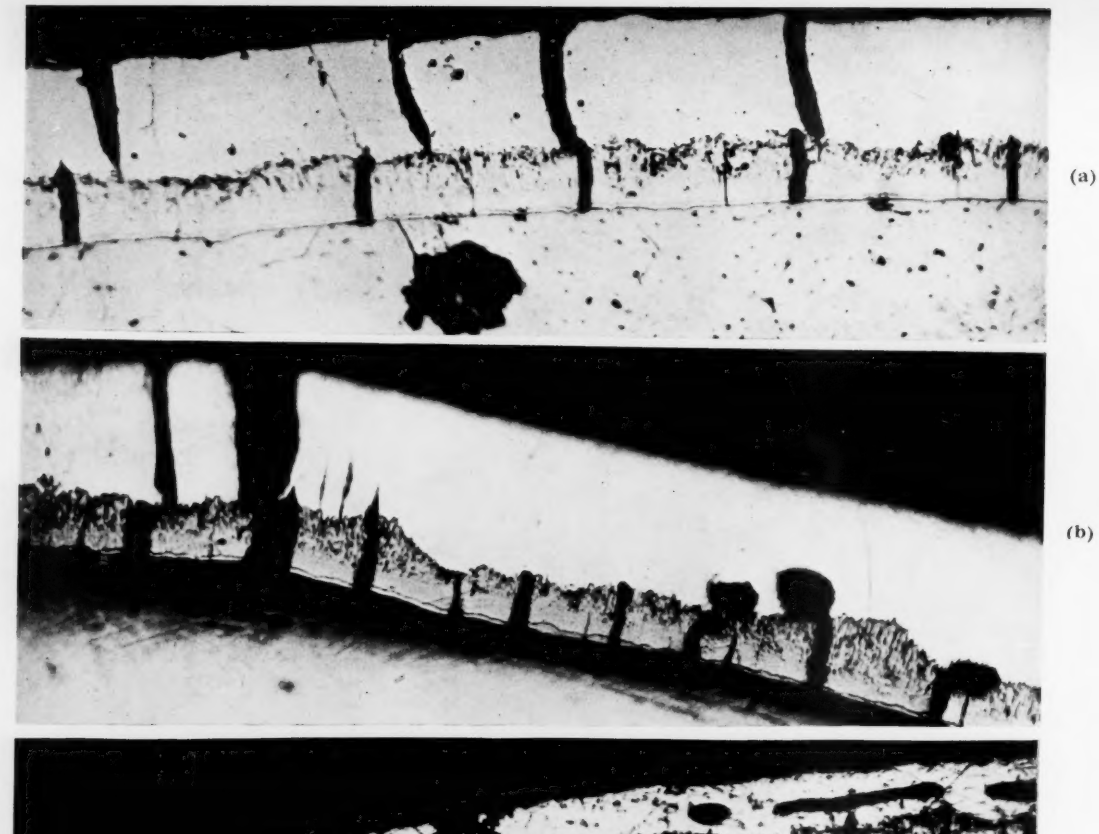


**Fig. 7—Comparison of adhesion of hot-galvanized coating to (*a*) gas-pickled, laboratory galvanized steel ; (*b*) sulphuric acid-pickled, laboratory galvanized steel ; (*c*) commercial galvanized steel ; (*d*) commercial galvanized steel, Sendzimir process** × 500

*Pearson, Bullough and Canning*]
*To face p.* 189]

there is a tendency for reaction rates to increase after prolonged treatment. This may be due to the fact that under these conditions the temperature of the now much-thinned strip is raised by the heat of reaction above that indicated by the thermocouple. Since long treatment times and marked thinning of steel would not be permitted in practice, this acceleration is not of great importance.

*Effect of Concentration of Hydrogen Chloride*—Figure 3*a* shows that, apart from concentrations of HCl below about 5%, loss in weight is directly proportional to concentration, as would be expected.

*Effect of Concentration of Carbon Dioxide*—The presence of $CO_2$ in the gas mixture increases the rate of attack on steel as compared with HCl, $N_2$ mixtures. The relative increase is greater at low than at high temperatures (*see* Figs. 1*a* and 5). Increasing proportions of $CO_2$, up to 10%, progressively increase reaction rates ; beyond this value increasing concentration has little further effect.

At high temperatures (Fig. 1*a*) and long treatment times (Fig. 4), the presence of $CO_2$ reduces the rate of attack as compared with HCl, $N_2$ mixtures. Figure 4 indicates that $CO_2$ prevents the acceleration which occurs in HCl, $N_2$ mixtures ; the reason for this and for the fact that, in the presence of $CO_2$, the temperature coefficient is less (*see* Fig. 1*a*), is not known.

*Effect of Gas-Flow Rate*—The rate of reaction between steel and HCl is directly proportional to the flow-rate of the gas mixture (Fig. 3*b*). This confirms that the reaction is diffusion-controlled.

Loss in weight is thus directly proportional to treatment time and gas velocity. In a furnace where steel strip is passed through an atmosphere containing a constant proportion of HCl at a fixed temperature, any increase in speed of strip will reduce treatment time but will correspondingly increase the velocity of the gas relative to the strip. If the gas velocity is small compared with that of the strip, the product of treatment time and relative gas velocity will remain essentially constant and the loss in weight per unit area of steel will also remain constant. This indicates why, in American practice, different strip speeds make no difference to the extent of pickling ; speeds are determined by the time required to reach pickling temperature in a pre-heating furnace.

*Nature of Attack on Steel Surface*—Figure 6 shows that the attack on a steel surface by gaseous HCl is entirely different from that by aqueous acid. In the latter, it is known that dissolution occurs by an electrochemical process, some portions of the surface acting as local anodes and others as cathodes, the anodic areas only being dissolved. On a steel surface which is essentially free from impurities, the local anodes and cathodes will be grains differing in orientation or in some other property. These areas of different electrode potential are clearly shown in Fig. 6*f* and *g*. At high temperatures in gaseous media, this type of corrosion is not possible. Instead, the corrosive gas, hydrogen chloride, attacks preferentially the most reactive areas (the grain boundaries in all the instances studied) in addition to causing a general uniform attack over the whole surface (*see* Fig. 6*a*, *b*, and *c*). Figure 6*e* clearly shows this intergranular penetration ; its absence with aqueous pickling is to be noted in Fig. 6*j*.

### Efficiency of Removal of High-Temperature Scale

The results reported above show that gaseous HCl at adequate concentration is quite efficient in removing scale. Because it is not possible to include inhibitors, any bare metal will suffer marked attack while scale elsewhere is being removed. In America the method is not used for descaling but solely for preparing a surface to receive a hot-dipped coating. The material used is cold-reduced strip, which is first pre-heated to pickling temperature in a separate furnace containing a slightly reducing atmosphere and then given a light etch in the gas-pickle before fluxing and galvanizing. Gas-pickling seems to be suitable only for steel which is ' bright ' or only very slightly oxidized. It might be said to correspond to the cracked ammonia stage in the Sendzimir galvanizing process.

### Formation of Carbon Monoxide

The presence of $CO_2$ in the gas-pickling medium causes an increase in the rate of attack at temperatures below 800° C. and with pickling times less than about 20 min., and produces CO in the exit gases. The primary reaction in gas pickling is :

$$Fe + 2HCl = FeCl_2 + H_2 \quad \ldots\ldots(1)$$

When carbon dioxide is present the following reactions are also possible :

$$H_2 + CO_2 = H_2O + CO \quad \ldots\ldots(2)$$

$$Fe + CO_2 = FeO + CO \quad \ldots\ldots(3)$$

$$FeO + 2HCl = FeCl_2 + H_2O \quad \ldots\ldots(4)$$

and it is seen that (2) and (3) can produce CO. It is not known what part $CO_2$ plays in accelerating the total reaction. It may be that the oxide formed by reaction (3) falls away as the steel under it is eaten away or that the primary reaction (1) can proceed at greater speed if the $H_2$ produced is removed from the surface by $CO_2$.

The concentration of CO in the exit gases is not sufficient to account for the increased loss in weight which occurs in the presence of $CO_2$. This is, however, explicable by considering that there is in the furnace a mixture of $CO_2$ and $H_2$ from the reaction of HCl with the steel. This mixture will be in equilibrium with CO and water according to the equation

$$CO_2 + H_2 \rightleftharpoons CO + H_2O \quad \ldots\ldots(5)$$

Any CO formed by reaction of $CO_2$ with steel will serve to move the equilibrium further to the left. By the time the gases leave the apparatus, *i.e.*, cool down to room temperature, the equilibrium will, on account of the cooling, have moved very much further to the left. The proportion of CO in the exit gases therefore depends mainly upon the rate at which the equilibrium (5) can adjust itself to the rapidly changing temperature conditions on cooling, and only to a slight extent upon the original CO concentration in the heated zone of the furnace. This explains why varying the temperature or $CO_2$ concentration makes no significant difference to the proportion of CO in the exit gases. The amount of CO is not such as to

constitute any toxicity danger, particularly as the waste gases would almost certainly be vented to the atmosphere along with other wastes.

**Adhesion of Galvanized Coatings**

The results given above show that gas-pickled surfaces produce more adherent galvanized coatings than do those pickled in aqueous $H_2SO_4$. The adhesion of these coatings to gas-pickled steel is of the same order as that of Sendzimir sheets, which are recognized as being of superior quality. This fact, linked with the micro-structure of the steel surfaces before galvanizing, fully confirms the claims made for the gas-pickling process. No completely satisfactory reason can be given for this enhanced adhesion. It might be considered that the intergranular attack of the pickling agent produces channels into which the coating can key ; on certain samples such penetration of the coating into the body of the steel can be seen when cross-sections are examined under the microscope. Good adhesion is, however, also obtained when the extent of pickling is small. In such a case, the layer of steel which has received the pickling treatment is all dissolved to form the iron–zinc alloy and no traces of keying can be seen. The steel surface after gas-pickling is cleaner than after sulphuric acid-pickling and this absence of smut may result in alloy layers of different structure.

A more probable reason seems to be that with slight gas-pickling, the surface of the steel is left essentially smooth, with slight attack at the more reactive boundaries. Under similar conditions $H_2SO_4$ etches away the surface of some grains, leaving others unattacked. This difference in surface may influence the structure of the alloy layers so that they possess a greater adhesion or, on bending, rupture in a different manner. It will be observed that the interface between outer zinc and alloy layers is almost plane in the case of gas-pickled steel and corrugated with $H_2SO_4$-pickled steel (*see* Fig. 7).

## CONCLUSIONS

The claim that gas-pickling produces a surface on steel which differs from that produced by aqueous acid-pickling and which is conducive to tighter hot-galvanized coatings is confirmed. The process has promise as a means of continuously treating steel strip or wire, and should avoid the production of large volumes of spent liquor. Because it involves heating to temperatures of the order of 700–750° C., it is suitable only for steels which do not require to be finished with a certain amount of cold work or special heat-treatment. Since the introduction of inhibitors into the pickling medium does not appear feasible, the process is suitable for removing only the thinnest of oxides, as non-uniform removal of scale will result in marked attack on the bare steel and so produce very uneven surfaces and heavy metal losses.

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# Determination of Tin in Highly Alloyed Steels

*By the Methods of Analysis Committee*

**SYNOPSIS**

A method of analysis is described which is suitable for the determination of tin in all types of steel. The method is primarily intended to cover the more highly alloyed steels which are outside the scope of the method (issued by the British Standards Institution) for plain and simple alloy steels.

The method depends upon solution of the steel in non-oxidizing acid, separation and removal of unattacked complex alloy carbides, followed by precipitation of tin as sulphide, together with molybdenum sulphide as a 'gathering' agent. The filtered sulphide precipitate is dissolved in mineral acid, and after adding the equivalent of 25 mg. of iron, the hydroxides of tin and iron are simultaneously precipitated with ammonia and the determination is completed by applying the conditions of the B.S.I. method to a solution of the precipitate.

The results show the performance of the method on high-speed tool steels and special types of high chromium–nickel steel containing alloying additions of cobalt, tungsten, molybdenum, titanium, niobium, etc. Various features affecting the quantitative isolation of tin from solutions containing these elements have been examined and are discussed. Attention has also been called to a statement by Scherrer, to the effect that, under certain conditions, tin could not be completely precipitated by hydrogen sulphide.

Consideration has been given to an alternative method involving complete solution of carbides, followed by sulphide precipitation from acid citrate solution, but this has marked limitations in the more highly alloyed ranges.

## Introduction

THE B.S.I. method[1] for the direct determination of tin is not applicable to highly alloyed steels. The Methods of Analysis Committee, recognizing the limitations of this method and the need for a more general procedure to cover, in particular, high-speed tungsten steels and other special classes of highly alloyed steels, directed that an investigation should be carried out with the object of covering this wider field.

The examination has been carried out by the Highly Alloyed Steels and Ferro-Alloys Sub-Committee, the constitution of which, during the period covered by the work, was as follows :

Mr. B. BAGSHAWE (*Chairman*) — Brown-Firth Research Laboratories

Paper MG/D/241/49 of the Methods of Analysis Committee of the Metallurgy (General) Division of the British Iron and Steel Research Association, received 9th January, 1950.

| | |
|---|---|
| Dr. J. CAMERON | Clyde Alloy Steel Co., Ltd. |
| Mr. W. T. ELWELL | I.C.I., Ltd., Billingham Division |
| Mr. L. E. GARDNER | Edgar Allen and Co., Ltd. |
| Mr. C. L. GRAYSON | English Steel Corporation, Ltd. |
| Mr. S. HARRISON | Kayser, Ellison and Co., Ltd. |
| Mr. J. D. HILL | Bragg Laboratory, N.O.I.D. |
| Mr. G. MURFITT | Wm. Jessop and Sons, Ltd. |
| Mr. R. T. POSTLETHWAITE | Samuel Fox and Co., Ltd. |
| Mr. J. L. WEST | Hadfields, Ltd. |

It was clear to the Sub-Committee that the requirements of this investigation could be met only by developing separation methods to isolate tin from interfering elements, following which the simple titration finish recommended in B.S. 1121 : Part 7 : 1948 could be employed. Attention was directed to a method for the determination of tin in high-speed steels,[2] in which complex carbides were separated and removed without decomposition, and the tin subsequently isolated by a sulphide precipitation.

Using this procedure as a basis, the work of the Sub-Committee has been directed along the following general lines :

(1) To confirm the adequacy of the procedure in the presence of tungsten, *e.g.*, for high-speed steels.

(2) To ascertain how far the principle of carbide-residue separation could be applied to other carbide-bearing types, *e.g.*, niobium steels.

(3) To obtain optimum conditions for the precipitation of stannic sulphide from a ferrous iron solution, with particular reference to the acidity range for complete precipitation and the avoidance of co-precipitation of the sulphides of other elements, *e.g.*, nickel and cobalt.

(4) To examine the effect of possible undecomposed organic tin compounds on the sulphide precipitation.

(5) To examine additional features which might invalidate the method, *e.g.*, loss of tin during the initial solution, interference of selenium, etc.

## HIGH-SPEED STEELS

Bagshawe and Dyke's method[2] has been applied to high-speed steels, but the Sub-Committee noted that removal of tungsten as unattacked carbide or tungstide was not always complete, and a substantial secondary hydrolysis of tungstic acid sometimes occurred after extracting the fumed concentrate derived from the solution of the sulphide precipitate. The addition of sulphurous acid during solution of the steel was of marked advantage in preventing oxidation and partial decomposition of the carbide residue. Results obtained by the Sub-Committee on four steels, covering a range of analyses of high-speed tool steel, are shown in Table I.

## APPLICATION TO OTHER HIGHLY ALLOYED STEELS

Four other steels with the compositions shown in Table V were chosen for further trials of the method. The series included types sufficiently complex to provide a thorough trial of the method in this wider field.

### Examination of Carbide Residues

Solution difficulties arose with steel MGS.60, and the elimination of interference by niobium is dealt with under a separate heading. The high-carbon samples responded in much the same way as high-speed steels, and no difficulty was experienced with the high-nickel sample at this stage. Carbide residues obtained from initial solution of the samples in hydrochloric acid were examined for occluded tin, after decomposition by the wet oxidation or alkaline fusion methods, as follows :

(*a*) The residue was ignited in a nickel crucible, fused with 5 g. of sodium peroxide, and the melt was extracted with water. The extract was boiled for a few minutes, 30 g. of tartaric acid were added, and the solution was acidified with sulphuric acid to give the equivalent of 2 % by volume of free sulphuric acid. The determination was then completed by sulphide precipitation and titration, as in the method.

(*b*) The residue, together with the filtering medium, was decomposed by wet oxidation, tartaric acid was added, and the solution was made ammoniacal. The solution was then acidified with hydrochloric acid to give the specified conditions for sulphide precipitation, and the determination continued from this point as in the method.

Many carbide residues were examined in this way, but in all cases the titre value was of the same order as that obtained from concurrently operated blank tests, and all titrations (inclusive of blank) were within 0·2 ml. of *N*/100 potassium iodate. From this, it is concluded that no significant amount of tin is retained by filtered carbide residues and that such

**Table I**

**DETERMINATION OF TIN IN HIGH-SPEED STEELS**

**Nominal Composition of Test Steels**

| Steel | W, % | Cr, % | V, % | Co, % | Mo, % | Cu, % |
|---|---|---|---|---|---|---|
| MGS.8 | 18 | 4 | 1 | ... | ... | ... |
| MGS.48 | 22 | ... | ... | 12 | ... | ... |
| MGS.49 | 18 | 4 | 1 | ... | ... | 0·5 |
| MGS.50 | 6¾ | 4½ | 1½ | 0·7 | 4 | ... |

**Results—Tin, % (Analysts A to I)**

| Steel | A | B | C | D | E | F | G | H | I | Average |
|---|---|---|---|---|---|---|---|---|---|---|
| MGS. 8 | 0·020 | 0·022 | 0·022 | 0·022 | 0·020 | 0·022 | 0·020 | 0·020 | 0·019 | 0·021 |
| MGS.48 | 0·044 | 0·041 | 0·045 | 0·041 | 0·044 | 0·041 | 0·043 | 0·042 | 0·041 | 0·042 |
| MGS.49 | 0·133 | 0·134 | 0·134 | 0·136 | 0·130 | 0·127 | 0·134 | 0·134 | 0·133 | 0·133 |
| MGS.50 | 0·039 | 0·041 | 0·041 | 0·040 | 0·040 | 0·039 | 0·039 | 0·039 | 0·039 | 0·039 |

residues may be rejected, without further examination, as a preliminary step to the separation of tin as sulphide.

**Niobium Steels : Prevention of Secondary Hydrolysis**

As indicated earlier, interference from niobium was experienced in preliminary tests on MGS.60. This was due to the fact that removal as carbide was incomplete and hydrolysis occurred in subsequent operations. During extraction of the fumed solution obtained after decomposition of the sulphide residue, precipitation of the hydrated acid might lead to loss of tin by adsorption. In certain instances, the niobium hydrolysis product was abnormally large. Further, secondary hydrolysis at this stage may not be quantitative, and residual niobium interferes at the final reduction stage owing to the development of dark-coloured reduction products which impair end-point detection. Further experience on this point showed that the end-point difficulty could be overcome by the use of a larger quantity of aluminium in the reduction, but the real problem remained, *viz.*, to eliminate secondary hydrolysis or to curtail it within limits where adsorptive loss of tin would not arise.

Attention was given to the possibility of preventing secondary hydrolysis at the sulphide stage by complex formation with tartrates and citrates. This failed, owing to buffering action, and co-precipitation of nickel and cobalt sulphides was induced. Stabilization of Group III elements as soluble tartaro-complexes did not appear to function satisfactorily in solutions not fully oxidized.

The possibility of removing niobium more completely at the carbide separation stage was then studied. Two aspects were important :

(*a*) Partial decomposition of carbides during solution, with the formation of soluble compounds which might not hydrolyse completely before filtration

(*b*) The physical nature of the carbides might be of significance, in that very finely disseminated carbides could become colloidal and pass the filter.

If co-precipitated with the sulphides, niobium remaining behind in this way would later appear as a precipitate after fuming with sulphuric acid. To assist hydrolysis of soluble niobium at the carbide-separation stage, the sulphurous acid addition was modified. This addition is now made in two stages, 10 ml. with the solvent acid, and 25 ml. on dilution.

The physical nature of the carbide appears to be affected by the ease or difficulty of completing solution of the sample, and is influenced by such factors as time and temperature of digestion. Complex steels may take several hours to dissolve in hydrochloric acid ; if the acid is heated, a finely dispersed carbide, likely to pass the filter or to suffer decomposition, may result.

It is recommended that very complex steels, *e.g.*, MGS.60, should be dissolved by digestion at a temperature not exceeding 60° C. Decomposition may be hastened initially by bringing the solution up to the boiling point, but thereafter low-temperature digestion should be used to complete solution. Steels of simpler compositions, including most of the austenitic niobium steels, may be dissolved more quickly, and emphasis on the vital nature of low-temperature digestion becomes less marked.

More complete separation of niobium is obtained if, after digestion, the diluted solutions are allowed to stand for several hours. In certain tests allowed to stand overnight, no trace of niobium was observed in the subsequent operations. The use of tight filter pads is an obvious auxiliary.

Results obtained on the niobium-bearing steel MGS.60 are shown in Table II. The results, which are in excellent agreement throughout, have not been affected, even in cases where the initial separation has been imperfect and minor amounts of niobium have been noted at subsequent stages. It is concluded, therefore, that there is some latitude in the amount of secondary hydrolysis which may occur without inducing significant adsorptive loss of tin, and that, provided the preliminary operations are conducted so that appreciable amounts of niobium

**Table II**

**TESTS ON 3½% NIOBIUM STEEL MGS.60**

| Method Variants | Remarks* | Tin, % |
|---|---|---|
| (1) Solution by recommended modified method | Nb separation as carbide complete<br>No evidence of secondary hydrolysis | 0·015, 0·015<br>0·016, 0·016 |
| (2) Solution as above, but digested at 80° C. for ½ hr. after dilution, cooled and filtered, instead of 2-hr. standing period | Nb separation complete<br>No secondary hydrolysis | 0·016 |
| (3) As (1), but with 1-hr. standing instead of 2 hr. | Separation complete<br>No secondary hydrolysis | 0·016 |
| (4) Dissolved at 90° C. instead of digestion at 60° C. | Carbide removal of Nb incomplete<br>Residual Nb (*a*) 30 mg., (*b*) trace | (*a*) 0·016<br>(*b*) 0·016 |
| (5) Dissolved at 90° C., otherwise as (3) | Residual Nb 13 mg. | 0·016 |
| (6) Dissolved at 90° C., otherwise as (1) | Residual Nb (*a*) 12 mg., (*b*) trace | (*a*) 0·016<br>(*b*) 0·016 |
| (7) Dissolved at 90° C. Allowed to stand overnight before filtration | No residual Nb | 0·016 |

*The quantities of niobium given in milligrammes may, in certain cases, include small amounts of tungsten hydrolysis product

do not survive the carbide-separation stage, subsequent secondary hydrolysis does not affect the accuracy of the determination.

Secondary hydrolysis is a feature also associated with steels of high titanium contents, but no interference has been observed during the later stages of the method. Titanium goes into solution during sulphuric acid fuming, and experimental evidence has shown that its presence during the final reduction in no way vitiates the results.

**Sulphide Precipitation**

Initial tests on the samples with high nickel and cobalt contents showed a tendency for co-precipitation of the sulphides of these elements, and when this occurred results were low and erratic. This was due to variations of practice in adjusting the acidity of the solution for sulphide precipitation.

Experiments have shown that quantitative precipitation of tin as sulphide can be obtained over a reasonably wide range of acidity. Results of tests using 2, 5, and 10 ml. of free hydrochloric acid (sp.gr. 1·16) in 450 ml. of solution volume for the sulphide precipitation are shown below :

*Results of Tin Determinations, Using Varied Acidity for Sulphide Precipitation*

| Free Acid in 450 ml. of Solution at Sulphide Precipitation, ml. of HCl (sp.gr. 1·16) | MG8.62 Sn, % | MG8.63 Sn, % |
|---|---|---|
| 2 | 0·029 | 0·022 |
| 5 | 0·031 | ... |
| 10 | 0·032 | 0·026 |

Where co-precipitation of nickel and cobalt sulphides has occurred, it appears to have been due to incorrect neutralization before adding the calculated excess of free acid; and by increasing the free acid to 10 ml. the additional latitude is such that co-precipitation of nickel and cobalt is completely prevented without effect on the separation of stannic sulphide or of the molybdenum sulphide carrier.

## EFFECT OF ORGANIC TIN COMPOUNDS ON THE SULPHIDE PRECIPITATION

The question of the effect of organic tin compounds on the sulphide precipitation arose from a reference to a statement by Scherrer,[3] that complete precipitation of tin as sulphide could not be obtained when the sample was dissolved in hydrochloric or sulphuric acids. Lundell, Hoffman, and Bright[4] accepted this statement, and recently it has been confirmed by Sayre and Reis.[5] More specifically, Scherrer indicated that material containing considerable amounts of carbon compounds decomposed by dilute non-oxidizing acids gave more trouble than material containing uncombined carbon or carbides insoluble in these acids. In particular, he referred to a 0·82% carbon steel which yielded only 50% of its tin content when dissolved in non-oxidizing acid and treated with hydrogen sulphide, whilst a normal tungsten–chromium–vanadium high-speed steel gave virtually a quantitative yield. To overcome the uncertainties attached to solution in non-oxidizing acids, Scherrer used nitric acid whenever possible, and he considered that the incompleteness of the sulphide precipitation might be due to the presence of stable organic tin compounds.

The wide circulation and general acceptance of Scherrer's views led to some misgivings as to the general application of the proposed method, which incorporates solution in non-oxidizing acids as a prelude to sulphide separation. Consequently, comparative tests were carried out on steels of the type said to be most likely to form organic tin compounds. Two carbon steels gave no significant variations in results when tested by three methods, including Scherrer's procedure, B.S. 1121 : Part 7 : 1948, and the proposed method. It was likely that the oxidation treatment with bromine water, prior to sulphide precipitation, incorporated in the proposed method, might be the reason for the good agreement inasmuch as it might destroy any organic tin compounds formed. In further tests, omitting the bromine treatment, however, the results again showed no variation.

So far no evidence had been produced of the presence of any tin compounds not responsive to sulphide precipitation, and it seemed likely that the reason for low yield lay in other directions. Tests were then made in which the molybdenum addition was omitted,

### Table III

**PRESENCE OF ORGANO-TIN COMPOUNDS : COMPARISON OF RESULTS BY VARIOUS METHODS**

| Method | Sn, % (Analyst A) | Sn, % (Analyst B) | Sn, % (Analyst C) |
|---|---|---|---|
| **Steel 32/852 (0·75% C)** | | | |
| (1) HCl solution, carbides removed | 0·039 | 0·039 | 0·037 |
| | 0·039 | 0·039 | |
| (2) B.S. 1121: part 7 | 0·041 | 0·041 | 0·041 |
| | 0·041 | 0·043 | |
| (3) Scherrer's method, $HNO_3$ solution | 0·037 | ... | 0·039 |
| | 0·037 | ... | |
| **Steel K2113 (0·66% C, 0·46% Cu)** | | | |
| (1) HCl solution, carbides removed | 0·197 | 0·198 | 0·200 |
| | 0·197 | 0·197 | |
| (2) B.S. 1121: part 7 | 0·205 | 0·209 | 0·200 |
| | 0·205 | 0·209 | |
| (3) Scherrer's method, $HNO_3$ solution | 0·194 | ... | 0·198 |
| | 0·195 | ... | |

### Table IV

**COMPARATIVE RESULTS OF TIN DETERMINATIONS ON PLAIN CARBON STEELS**

| HCl Solution, Carbide-Removal Method : Sn, % | | | | $HNO_3$ Solution, Scherrer Method : Sn, % |
|---|---|---|---|---|
| With Br With Mo | With Br Without Mo | Without Br With Mo | Without Br Without Mo | |
| **Steel K2113 (0·66% C, 0·46% Cu)** | | | | |
| 0·198 | 0·198 | 0·201 | 0·199 | 0·200 |
| 0·200 | 0·198 | 0·201 | 0·201 | 0·202 |
| **Steel 24/9514 (0·80% C, 0·15% Cu)** | | | | |
| 0·044 | 0·045 | 0·044 | 0·044 | 0·044 |
| 0·045 | 0·045 | 0·045 | 0·045 | 0·046 |
| **Steel W1031 (1·32% C, 0·11% Cu)** | | | | |
| 0·016 | 0·017 | 0·016 | 0·016 | 0·017 |
| 0·017 | 0·016 | 0·016 | 0·017 | 0·017 |

both with and without prior bromine treatment, but again no low yields were observed. The results, shown in Tables III and IV, furnish ample evidence of the completeness of sulphide separation under the conditions specified in the proposed method (Appendix I).

## POSSIBILITY OF LOSS OF TIN DURING SOLUTION

The object of this work was to ascertain whether any tin was lost by volatilization. Six samples of MGS.49 (0·135% Sn) were placed in 500-ml. Taylor-pattern flasks, fitted with a bent tube leading through a bung into a second flask, with the tip of the bend just under a small quantity of water. This second flask carried a 30-in. reflux condenser. Hydrochloric acid was added and the solutions were boiled continuously for 8 hr. The acid content of the condensate was then adjusted to that specified in the method. After reduction by antimony/aluminium, all samples gave a titre of 0·2–0·3 ml. of $N/100$ potassium iodate. Blank tests, operated simultaneously, gave the same, or closely similar, titres, and experiments with alternative methods of collecting the volatile products produced comparable results. The volatilization of tin at this stage is therefore negligible.

## SELENIUM-BEARING STEELS

The possible interference of selenium has been examined on two selenium steels and also on pure solutions. Selenium is evolved as hydrogen selenide during solution of the steel in hydrochloric acid, and in boiling solutions there is little doubt that expulsion of the element is complete. When the period of boiling is restricted and the steel must be dissolved by digestion at 60° C., the filtered carbide residue smelt strongly of hydrogen selenide, and the influence of the element on the aluminium/antimony reduction and iodate titration was therefore tested.

The conditions of the reduction stage were simulated by preparing a solution containing 130 ml. of water and 70 ml. of hydrochloric acid (sp.gr. 1·16) to which were added the equivalents of 0·025 g. of iron and 0·2% and 0·5% of selenium. The solutions were reduced with antimony and aluminium, as in the normal method. The average consumption of $N/100$ potassium iodate was only 0·15 ml., the same, or closely similar, values being obtained in selenium-free blank tests.

The aluminium addition results in the formation of hydrogen selenide which is evolved with a characteristic odour. It seems certain, therefore, that even if some selenium survives the initial stages of the process, complete removal of residual amounts occurs at the aluminium-reduction stage and the titrations are unaffected.

Results obtained on two selenium steels are shown below :

*Results of Tin Determinations on Selenium Steels*

| Sample 540 (0·24% Se) | | Sample 542 (0·18% Se) | |
|---|---|---|---|
| Solution at B.P. | Solution at 60° C. | Solution at B.P. | Solution at 60° C. |
| 0·011 | 0·011 | 0·015 | 0·015 |
| 0·011 | 0·011 | 0·015 | 0·014 |
| 0·011 | 0·012 | 0·015 | 0·015 |
| 0·014 | 0·014 | 0·015 | 0·015 |
| 0·011 | 0·010 | 0·014 | 0·015 |
| 0·010 | 0·010 | 0·015 | 0·015 |
| 0·010 | ... | 0·015 | ... |

## RANGE

To confirm the applicability of the method for the determination of tin at higher levels, three members analysed a specially made complex alloy steel. Results on 2·5-g. and 5-g. sample weights were in excellent agreement, and although a range of only 0–0·25% of tin is claimed, the method could undoubtedly be used with equal confidence up to at least 0·5% of tin, if necessary.

## AN ALTERNATIVE METHOD

Consideration has been given to an alternative method (Appendix II) for the determination of tin in highly alloyed steels and, in fact, much experimental work has been carried out with this procedure on the steels already mentioned. Originally intended only for high-speed steels, the method incorporates solution of the sample in hydrochloric acid, followed by digestion with nitric acid to effect decomposition of carbides ; tungsten is held in solution by the use of citrate or tartrate during the sulphide precipitation of tin, and thereafter the method follows the lines

**Table V**

**DETERMINATION OF TIN IN COMPLEX ALLOY STEELS**

**Nominal Composition of Test Steels**

| Steel | C, % | Ni, % | Cr, % | Co, % | W, % | Nb, % | Ti, % | Mo, % | V, % | Zr, % |
|---|---|---|---|---|---|---|---|---|---|---|
| MGS.60 | ... | 13½ | 13½ | 10 | 3 | 3½ | ... | 2 | ... | ... |
| MGS.61 | 2¼ | ... | 14 | ... | ½ | ... | ... | ... | ... | ... |
| MGS.62 | 2¼ | 2 | 18 | 2 | 2 | ... | ... | ... | ... | ... |
| MGS.63 | Low | 30 | 20 | ... | ... | ... | 1¼ | ... | ... | ... |
| MGS.120 | Low | 7 | 18 | ... | 3 | 1½ | 1 | ... | 1 | ½ |

**Results—Tin, %-(Analysts A to H)**

| Steel | A | B | C | D | E | F | G | H | Average |
|---|---|---|---|---|---|---|---|---|---|
| MGS.60 | 0·014 | 0·014 | 0·015 | 0·016 | 0·015 | 0·015 | 0·016 | 0·016 | 0·015 |
| MGS.61 | 0·015 | 0·015 | 0·015 | 0·016 | 0·018 | 0·020 | 0·016 | 0·016 | 0·016 |
| MGS.62 | 0·029 | 0·026 | 0·027 | 0·027 | 0·030 | 0·026 | 0·031 | 0·029 | 0·028 |
| MGS.63 | 0·026 | 0·023 | 0·025 | 0·023 | 0·026 | 0·026 | 0·026 | 0·025 | 0·025 |
| MGS.120 | 0·245 | ... | ... | ... | 0·247 | ... | ... | 0·248 | 0·247 |

of the standard procedure. Provision for the use of molybdenum as a carrier was included.

For samples containing carbides which are not difficult to decompose, the method has much to recommend it. Excellent results were obtained on MGS.63, for example, without in any way departing from the suggested procedure. Steels MGS.61 and MGS.62, however, contain large amounts of chromium carbides which were difficult to decompose and, if the method was carried forward without complete solution, the carbides broke down in the subsequent sulphuric acid fuming stage and the end-point of the final titration was obscured by a dark-green coloration due to chromium salts. To overcome this difficulty two procedures were included experimentally in the method.

(*a*) A persulphate oxidation treatment was given to the solution obtained after decomposition of the sulphide precipitate, thereby converting the chromium to chromate, prior to the precipitation of tin and iron with ammonium hydroxide

(*b*) A solution of the sample in sulphuric acid (1 : 4) was evaporated to fuming to break up the carbides completely.

Good results were obtained with either modification, but (*b*) presented practical difficulties with large sample weights.

The niobium-bearing sample combined the carbide-decomposition problem with the niobium hydrolysis previously discussed, and the results, when modified treatments were given, tended to be low and variable.

In view of the obvious difficulties of applying this procedure to the more highly alloyed types, the examination was discontinued. However, the principle of citrate complex formation might be useful in the development of future applications to ferro-tungsten.

## RESULTS AND CONCLUSION

The recommended method is described in Appendix I, and results obtained by the method on a selection of very complex steels are shown in Table V. The results demonstrate the wide scope and reproducibility of the method in its application to these special types of highly alloyed steel.

## References

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## Appendix I

### TENTATIVE METHOD FOR DETERMINATION OF TIN IN HIGHLY ALLOYED STEELS (INCLUDING HIGH-SPEED STEEL)

#### Introduction

*Principle*—The method depends upon removal of the carbide residue from an initial solution of the steel in a non-oxidizing acid. This is followed by isolation of tin as the sulphide, using molybdenum sulphide as 'carrier' agent, solution of the sulphide, reduction of tin to the bivalent condition with metallic aluminium in the presence of an antimony salt, and titration of the reduced tin with standard iodate.

*Range*—Up to 0·25% Sn.

*Reproducibility*—With 0·05% Sn present, ±0·003% Sn ; with 0·20% Sn present, ± 0·006% Sn.

#### Special Apparatus Required

A suitable assembly for reduction of the tin consists of a 350-ml. conical flask, marked at 200 ml. and having a neck of about 1-in. dia. The flask is fitted with a bung, holding a glass syphon tube of about $\frac{1}{4}$-in. bore, which protrudes about $\frac{1}{2}$ in. into the flask below the base of the bung and externally is bent so as to overhang the edge of the hot plate. Grade *A* graduated glassware shall be used.

#### Reagents Required

All reagents shall be of the highest purity obtainable, and distilled water shall be used throughout. Solutions shall be freshly prepared and, where necessary, filtered. Details of the solutions required are as follows :

*Hydrochloric Acid* (1 : 19)—Dilute 50 ml. of hydrochloric acid (sp.gr. 1·16) to 1 litre with water.

*Sulphurous Acid*—Pass sulphur dioxide gas into water until a saturated solution is obtained.

*Bromine Water*—To 500 ml. of water add bromine, 1 ml. at a time, with vigorous shaking after each addition, until a few drops of bromine remain undissolved on the bottom of the flask ; 10 ml. of bromine should be ample for saturation. This solution should be prepared under a good draught.

*Ammonium Hydroxide* (1 : 1)—Dilute 500 ml. of ammonium hydroxide (sp.gr. 0·880) to 1 litre.

*Ammonium Molybdate* (1%)—Dissolve 1 g. of ammonium molybdate crystals ($(NH_4)_6Mo_7O_{24}.4H_2O$) in water and dilute to 100 ml.

*Acid Sulphide Wash*—To about 900 ml. of water, add 10 ml. of sulphuric acid (sp.gr. 1·84), dilute to 1 litre and pass hydrogen sulphide gas through the solution until saturated.

*Ferric Chloride*—Dissolve 0·5 g. of pure tin-free iron in 20 ml. of hydrochloric acid (sp.gr. 1·16) and oxidize with the minimum amount of nitric acid (sp.gr. 1·42), added dropwise. Boil to expel nitrous fumes, cool, and dilute to 100 ml.

*Hydrochloric Acid* (1 : 1)—Dilute 500 ml. of hydrochloric acid (sp.gr. 1·16) to 1 litre.

*Antimony Chloride*—Treat 0·5 g. of powdered antimony with 3 ml. of sulphuric acid (sp.gr. 1·84) and fume for 5 min. Cool, dissolve in a mixture of 20 ml. of hydrochloric acid (sp.gr. 1·16) and 50 ml. of water, boil for 5 min., cool, and dilute to 100 ml.

*Sodium Bicarbonate* (10%)—Dissolve 100 g. of sodium bicarbonate in water* and dilute to 1 litre.

*Potassium Iodide* (10%)—Dissolve 10 g. of potassium iodide in water* and dilute to 100 ml. This solution must be freshly prepared and free from iodate.

* Solutions of bicarbonate, iodide, starch, and iodate, should be as free from dissolved oxygen as practicable. It is therefore necessary to use freshly boiled distilled water for these solutions.

*Starch*—Make a suspension of 4 g. of powdered starch in 50 ml. of water and add carefully to 450 ml. of boiling water. Cool before use. This solution must be freshly prepared.*

*Potassium Iodate* (N/100)—Dissolve 0·3567 g. of potassium iodate in water* and dilute to exactly 1 litre.

**Sampling**

Recommended methods of obtaining a suitable sample for the analytical procedure outlined below are described in B.S.....†

**Procedure**

Transfer 5 g. of sample to a 600-ml. tall beaker, add 60 ml. of hydrochloric acid (sp.gr. 1·16), 10 ml. of sulphurous acid, and digest (*see* Note 1, below), avoiding appreciable evaporation, until the metal is dissolved and only a residue of carbide remains undecomposed. Add 100 ml. of hot water and 25 ml. of sulphurous acid. Add a little ashless paper pulp and boil for 5 min. Set aside for a few minutes (*see* Note 2) to allow the carbide residue to settle. Filter the solution through a tight pulp pad, by decantation as far as possible, and wash the residue with hot hydrochloric acid (1 : 19) containing a few drops of sulphurous acid. Discard the carbide residue.

To the filtrate add 5 ml. of bromine water and boil for a few minutes. Cool and neutralize the solution carefully with ammonium hydroxide (1 : 1) until a faint permanent hydroxide precipitate is just obtained. Just discharge this precipitate with hydrochloric acid (sp.gr. 1·16), added dropwise, and add 10 ml. excess.

Dilute the solution to about 450 ml., heat to boiling, add 2 ml. of ammonium molybdate (1%), and then pass a brisk stream of hydrogen sulphide through the solution for 1 hr. Allow to stand (preferably overnight), filter through a pulp pad, and wash the precipitated sulphides with sulphide wash.

Return the pad and precipitate to the original beaker and destroy paper, etc., by adding 25 ml. of nitric acid (sp.gr. 1·42) and 10 ml. of sulphuric acid (sp.gr. 1·84) and evaporating to fumes of sulphur trioxide, adding a further 10 ml. of nitric acid and repeating the evaporation (Note 3). Extract the fumed solution with 100 ml. of water and 15 ml. of hydrochloric acid (sp.gr. 1·16), and heat to boiling. A small amount of tungstic acid may precipitate at this stage, and in this event filter the solution through a pulp pad and wash with hydrochloric acid (1 : 19).

Add 5 ml. of ferric chloride solution, add ammonium hydroxide (1 : 1) cautiously to the boiling liquid until the solution is alkaline, filter the precipitated hydroxides of iron and tin through a No. 541 paper, and wash with hot water. Dissolve the precipitate by pouring 140 ml. of hot hydrochloric acid (1 : 1) through the paper, collecting the solution in a flask (*see* " Special Apparatus Required "), and finally wash several times with hot hydrochloric acid (1 : 19).

Add to the solution 2 ml. of antimony chloride solution, dilute to 200 ml. with water, and place on a hot plate (Note 4). When nearly boiling, add carefully tin-free aluminium (millings, drillings, or foil) in small amounts until approximately 1 g. has been added. To the neck of the flask, before final solution of the last addition of aluminium, fit a bung with bent delivery tube which overhangs the edge of the hot plate. Bring to the boil and continue boiling for 30 min., allowing the condensate to drip into a beaker for discard. After 30-min. boiling, immediately immerse the end of the delivery tube in 200 ml. of sodium bicarbonate solution (10%) contained in a tall-form 300-ml. beaker. Allow to suck back and evolve carbon dioxide, and then cool the flask in running water, keeping the end of the delivery tube immersed until quite cold (15–20° C.).

Remove the bung, add 10 ml. of potassium iodate solution (10%) and 20 ml. of starch solution. Titrate with potassium iodate solution (*N*/100) to a permanent blue colour, using a 10-ml. or 25-ml. burette of not more than 8-mm. internal dia.

**Calculation**

1 ml. of potassium iodate (*N*/100) is equivalent to 0·0005935 g. of tin, or 0·0119% of tin on a 5-g. sample.

**Notes**

(1) Certain types of very highly alloyed steels dissolve very slowly in the hydrochloric acid. If these steels contain elements such as niobium, tungsten, or titanium, the solution process should be carried out by hot digestion at a temperature not exceeding 65° C. The solution may finally be heated to boiling for a minute or so to ensure that solution is complete.

(2) For niobium-, tungsten-, or titanium-bearing steels, set aside for 2 hr.

(3) Oxidation and fuming must be repeated until all organic matter is destroyed.

(4) A blank determination should be made under the same conditions, using tin-free bar iron or steel.

## Appendix II

### ALTERNATIVE METHOD FOR DETERMINATION OF TIN IN HIGH-SPEED STEEL AND FERRO-TUNGSTEN

**Introduction**

*Principle*—The sample is dissolved in acid, the solution oxidized, and the precipitated tungstic acid redissolved by the addition of ammonia, iron precipitation being prevented by adding citric acid. The solution is re-acidified and the tin is isolated as the sulphide, using molybdenum sulphide as 'carrier.' The sulphide is separated and redissolved, the tin is reduced to the bivalent condition with metallic aluminium in the presence of an antimony salt, and the reduced tin is titrated with standard iodate solution.

*Range*—Not established.

*Reproducibility*—Not established.

**Special Apparatus Required**

A suitable assembly for reduction of the tin consists of a 350-ml. conical flask, marked at 200 ml. and having a neck of about 1-in. dia. The flask is fitted

* Solutions of bicarbonate, iodide, starch, and iodate should be as free from dissolved oxygen as practicable. It is therefore necessary to use freshly boiled distilled water for these solutions.

† In course of preparation.

with a bung holding a glass syphon tube of about ¼-in. bore, which protrudes about ½ in. into the flask below the base of the bung and externally is bent so as to overhang the edge of the hot plate. Grade *A* graduated glassware shall be used.

**Reagents Required**

All reagents shall be of the highest purity obtainable, and distilled water shall be used throughout. Solutions shall be freshly prepared and, where necessary, filtered. Details of the solutions required are as follows :

*Ammonium Citrate* (60%)—Dissolve 500 g. of citric acid ($H_3C_6.H_5O_7.H_2O$) in 500 ml. of ammonium hydroxide (sp.gr. 0·880), cool, and dilute to 1 litre.

*Sulphuric Acid* (1 : 4)—To 700 ml. of water cautiously add 200 ml. of sulphuric acid (sp.gr. 1·84), mix, cool, and dilute to 1 litre.

*Ferric Sulphate* (0·5%)—Dissolve 5 g. of ferric sulphate ($Fe_2(SO_4)_3$) in 50 ml. of hydrochloric acid (sp.gr. 1·16), and dilute to 1 litre.

*Hydrochloric Acid* (1 : 19)—Dilute 50 ml. of hydrochloric acid (sp.gr. 1·16) to 1 litre with water.

*Ammonium Hydroxide* (1 : 1)—Dilute 500 ml. of ammonium hydroxide (sp.gr. 0·880) to 1 litre.

*Ammonium Molybdate* (1%)—Dissolve 1 g. of ammonium molybdate crystals ($(NH_4)_6.Mo_7O_{24}.4H_2O$) in water, and dilute to 100 ml.

*Acid Sulphide Wash*—To about 900 ml. of water, add 20 ml. of sulphuric acid (sp.gr. 1·84), dilute to 1 litre, and pass hydrogen sulphide gas through the solution until saturated.

*Hydrochloric Acid* (1 : 1)—Dilute 500 ml. of hydrochloric acid (sp.gr. 1·16) to 1 litre.

*Antimony Chloride*—Treat 0·5 g. of powdered antimony with 3 ml. of sulphuric acid (sp.gr. 1·84) and fume for 5 min. Cool, dissolve in a mixture of 20 ml. of hydrochloric acid (sp.gr. 1·16) and 50 ml. of water, boil for 5 min., cool, and dilute to 100 ml.

*Sodium Bicarbonate* (10%)—Dissolve 100 g. of sodium bicarbonate in water,* and dilute to 1 litre.

*Potassium Iodate* (10%)—Dissolve 10 g. of potassium iodate in water,* and dilute to 100 ml. This solution must be freshly prepared and free from iodate.

*Starch*—Make a suspension of 4 g. of powdered starch in 50 ml. of boiling water. Cool before use. This solution must be freshly prepared.*

*Potassium Iodate* (*N*/100)—Dissolve 0·3567 g. of potassium iodate in water,* and dilute to exactly 1 litre.

**Sampling**

Recommended methods of obtaining a suitable sample for the analytical procedure outlined below are described in B.S.....†

**Procedure**

Weigh out 5 g. of sample into a 600-ml. tall beaker, and add 60 ml. of hydrochloric acid (sp.gr. 1·16). Allow the solution to digest for 20–30 min. Oxidize with nitric acid, and continue digestion until all tungstic acid present is clean. Dilute to 75 ml. with water, remove from the source of heat, and whilst still warm add 100 ml. of ammonium citrate (60%), add ammonium hydroxide (sp.gr. 880) until ammoniacal, and then 10 ml. excess to take any tungstic acid into solution. Warm if necessary to complete solution. Neutralize with sulphuric acid (1 : 4), add 40 ml. excess, and dilute to 400 ml.

Add 2 ml. of ammonium molybdate solution (1%), heat the solution to about 70° C., and pass a rapid stream of hydrogen sulphide for 45 min. Allow the precipitate to settle overnight, and filter through a pulp pad, washing with acid sulphide wash. Discard the filtrate. Transfer the pad to the original beaker, wipe the funnel with filter paper, and add that also to the beaker. Add 10 ml. of water, 10 ml. of sulphuric acid (sp.gr. 1·84), and 15 ml. of nitric acid (sp.gr. 1·42).

Place on the hot plate and heat until the pulp is destroyed. Should a brown coloration occur, indicating incomplete destruction of the pad, remove the beaker from the plate and carefully add further nitric acid. The solution is finally evaporated to fumes and fumed for 5 min.

Remove from the plate, cool, dilute to 50 ml., and filter to remove the sulphur globules. To the filtrate add 10 ml. of ferric sulphate solution. Precipitate the ferric and stannic hydroxides with ammonia, adding 1–2 ml. excess. Warm to coagulate the precipitate, and filter through a pulp pad, washing with hot water.

Dissolve the precipitate with 140 ml. of hot hydrochloric acid (1 : 1), collecting the solution in a flask (*see* "Special Apparatus Required"), and finally wash several times with hot hydrochloric acid (1 : 19).

Add to the solution 2 ml. of antimony chloride solution, dilute to 200 ml. with water, and place on a hot plate.* When nearly boiling, carefully add tin-free aluminium (millings, drillings, or foil) in small amounts until approximately 1 g. has been added. To the neck of the flask, before final solution of the last addition of aluminium, fit a bung with bent delivery tube which overhangs the edge of the hot plate. Bring to the boil and continue boiling for 30 min., allowing the condensate to drip into a beaker for discard. On completion of 30-min. boiling, immediately immerse the end of the delivery tube under 200 ml. of sodium bicarbonate solution (10%) contained in a tall 300-ml. beaker. Allow to suck back and evolve carbon dioxide, and then cool the flask in running water, keeping the end of the delivery tube immersed until quite cold (15–20° C.).

Remove the bung and add 10 ml. of potassium iodide solution (10%) and 20 ml. of starch solution. Titrate with potassium iodate solution (*N*/100) to a permanent blue colour, using a 10-ml. or 25-ml. burette of not more than 8-mm. internal dia.

**Calculation**

1 ml. of potassium iodate (*N*/100) is equivalent to 0·0005935 g. of tin, or 0·0119% of tin on a 5-g. sample.

* Solutions of bicarbonate, iodide, starch, and iodate should be as free from dissolved oxygen as practicable. It is therefore necessary to use freshly boiled distilled water for these solutions.

† In course of preparation.

* A blank determination should be made under the same conditions, using tin-free bar iron or steel.

# The Reduction of Lump Ores

*By R. Wild, Ph.D., and H. L. Saunders, Ph.D., F.R.I.C.*

**SYNOPSIS**

This paper deals with the overall rate of heat transfer to the centre of ore lumps and the penetration of gaseous reduction. The effect of various factors upon the rate of heat transfer has been assessed, and the temperature lag at the centre of lumps heated at a uniform rate is related to their size. Specimens of a wide range of ores have been subjected to reduction by blast-furnace gas under standardized conditions and subsequently the oxygen gradient from the surface to the centre has been found by chemical analysis. The extent of the penetration of reduction has been related to the porosity of the ore which is, in general, the factor limiting reduction within the lump.

## Introduction

For the most efficient operation of a blast-furnace the ore charged should be of a suitable size. If it contains a high proportion of fines, channelling and excessively high dust losses limit the blowing rate; on the other hand, large masses of uncrushed stone will proceed down the stack with insufficient preparation taking place within the lump. Since the physical and chemical properties of ores vary greatly, there can be no one optimum size applicable to all ores, but it should be possible to size each ore so that the lumps reach the hearth in an equally prepared state.

In the upper part of the furnace, water is driven off the ore and, in the case of carbonate ores, some of the $CO_2$. This is beneficial in so far as it opens up the lump to subsequent penetration by reducing gases, but, if the heating rate is high, large lumps may be subjected to high internal gas pressure with consequent disintegration.

The problem of the correct sizing of ores is thus two-fold, firstly to obtain lumps of such a size that the heating rate will neither produce disintegration with subsequent dust losses nor give inadequate heating at the centre; and, secondly, to obtain the correct lump size so that gas penetration into the lump will be sufficient to effect adequate chemical reduction during the descent of the stack to prevent the hearth being chilled by endothermic reducing reactions. These two conditions may lead to conflicting values and it may be necessary to balance the one against the other, or the effect of one may be overriding, thus making it possible to neglect the other.

The importance of adequate heat flow and gas penetration into the lump, and thus the necessity for the correct sizing of the ore, has been emphasized by many workers, including Colclough,[1] Joseph,[2, 3] and Lewis,[4] whilst the difference in operation of an experimental blast-furnace consequent upon a variation of ore size was found by Royster, Joseph, and Kinney.[5]

## HEAT TRANSFER—PRELIMINARY SURVEY

A preliminary survey of the factors influencing heat transfer through the lump was carried out in a simple manner by placing thermocouples at the surface and at the centre of the desired specimens which were then inserted into a suitable furnace preheated to the experimental temperature. The disadvantage of this simple procedure is that in cases where the ore contains moisture or readily evolved $CO_2$, fissuring of the sample leads to lack of reproducibility of results to an extent which, in extreme cases, may overshadow the effect of all other variables. These effects may be removed by drying the sample before use, but the results obtained after such pretreatment, although valuable for comparative purposes, do not give a true picture of the behaviour of these ores in normal practice. Cracking has been minimized in certain cases by heating in two stages, room temperature to 540° C., and 540° to 900° C. From a number of experiments the figures from samples showing marked disintegration at the close of the experiment were eliminated, and thus the results may be regarded as more typical of the unbroken ores.

The factors studied are considered under the following headings:

Nature of material
Moisture content
Shape
Size
Degree of reduction.

The ores used were Sorpresa Rubio, Zaccar, Grangesberg, Oxfordshire, Northamptonshire, Frodingham, Eaton, and Sproxton, and, in addition, limestone.

### EXPERIMENTAL PROCEDURE

Essentially the same experimental procedure was employed throughout this preliminary work. A small hole, normally $\frac{1}{16}$ in., and always smaller than $\frac{1}{8}$ in., was drilled to the centre of the selected specimen, and into this hole a fine Pt/13% Pt–Rh thermocouple was inserted, the wires being packed around with the drillings and so sealed as far as possible against abnormal gas ingress at this point. The sample was inserted into a furnace already at the required temperature, measured by a second thermocouple at

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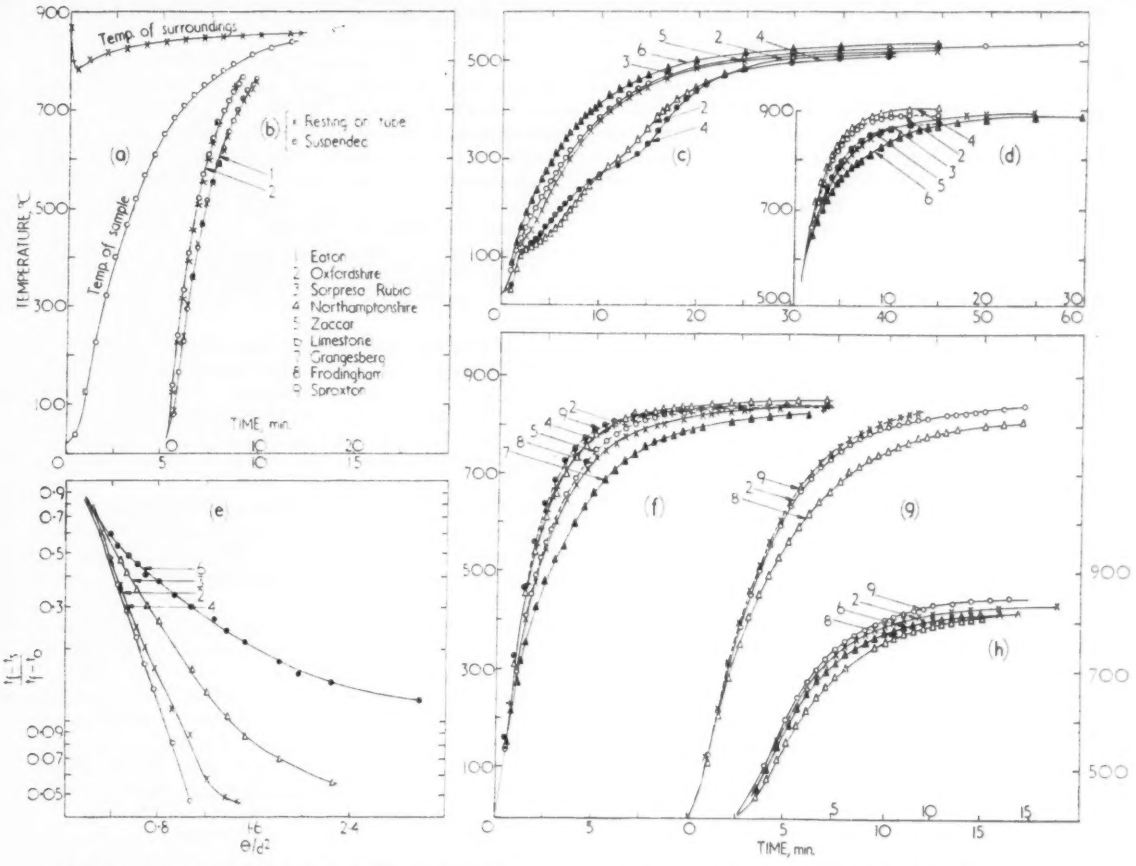



(*a*) Changes in temperature of surroundings during heating of sample
(*b*) Comparison of rates of heating of suspended and unsuspended samples
(*c*) Various undried samples (room temp. to 540° C.), $l = 7$, $d = 2\frac{1}{2}$ cm.
(*d*) Various samples (540° to 900° C.), $l = 7$, $d = 2\frac{1}{2}$ cm.
(*e*) Rectified rate-of-heating curves
(*f*) Dried ores (room temp. to 870° C.), $l = 4$, $d = 2$ cm.
(*g*) Dried ores (room temp. to 870° C.), $l = 7$, $d = 2\frac{1}{2}$ cm.
(*h*) Dried ores (435° to 870° C.), $l = 7$, $d = 2\frac{1}{2}$ cm.

**Fig. 1—Curves of rate of heating of ore under various conditions**

the centre of the hot zone. From continuous readings of these two couples, curves for the rate of heating were obtained.

Three tube furnaces, *A*, *B*, and *C*, were employed, having diameters of 2·7, 4·5, and 11·3 cm., respectively. Furnaces *A* and *B*, which had very similar heat capacities, were used for the major portion of the work. Although no exactly similar experiments were carried out in these two furnaces, reference to Figs. 1*d* and *f* (units *A* and *B*), shows that the relative positions of the three ores common to each are the same in both cases. Furnace *C* was used solely for the assessment of the effect of lump size.

For the direct comparison of the rate of temperature rise at the centre for different specimens, the most satisfactory arrangement would be to use an infinitely large heat source of constant temperature which was also infinitely conducting; the furnace temperature would then remain constant throughout the experiment. With the best experimental approach, however, there is inevitably a variation of this temperature with time, the extent of which is shown under typical conditions in Fig. 1*a*, to be small over the main portion of the heating range.

It was thought that the manner of supporting the specimen in the furnace tube might lead to variations in the heating rate. Thus two comparative tests were carried out, the specimen in one resting on the furnace tube and in the other being supported co-axially with the tube on very thin mica washers. Figure 1*b* shows that this causes no appreciable difference in the heating rate and thus the former and simpler method was adopted throughout.

### Nature of Material

The rate of heating, which throughout this section is expressed as the rate of rise of temperature at the centre of the specimen, of seven different ores and limestone was found by the experimental procedure just described. The specimens were cylindrical and 7 cm. long × $2\frac{1}{2}$ cm. in dia., with one exception where the dimensions were 4 cm. × 2 cm. The effects of moisture were eliminated in all cases except one by pre-drying the specimens.

Figures 1*c*, *d*, *f*, *g*, and *h* show these rates of heating. Figures 1*c* and *d* refer to the two-stage heating of the same specimens, Figs. 1*f* and *g* to the rates of heating of specimens over the whole range in a single stage, and Fig. 1*h* to heating between 430° and 870° C.

Neglecting, for the present, the effect of moisture, there is no very great difference between the rate of heating of any of these ores. They can, however, be placed in a definite order, the rate of temperature rise at the centre decreasing in the following scale: Sproxton (dry) > Northamptonshire (dry) > Oxfordshire (dry) > Zaccar > Sorpresa Rubio > Frodingham (dry) > Grangesberg. The Grangesberg used for this work was a very low-grade specimen; other work, using a high-grade magnetite (Kiruna $C_1$) showed that this was a much better conductor of heat.

Figure 1*e* shows the rectified rate-of-heating curves, *i.e.*:

$$\frac{t_f - t_s}{t_f - t_o}$$

plotted against $\theta/d^2$ on a log arithmetic scale, where

$t_f$ = temperature of surroundings (*i.e.*, furnace temperature)
$t_s$ = temperature of centre of specimen
$t_o$ = initial temperature
$\theta$ = time in minutes
$d$ = diameter of specimen

obtained from the data plotted in Fig. 1*d*.

If $k$ (thermal conductivity), $H_T$ (heat-transfer coefficient from the surroundings to the surface of the sample), $k/\rho cp$ (thermal diffusivity (where $\rho$ = density and $cp$ = specific heat)), and $t_f$ are constant throughout the experiments, then the curves should be straight lines over all except a short initial portion. It has already been noted (Fig. 1*a*) that $t_f$ is almost constant, except over a short initial period. The curves, with the exception of that relating to limestone, are, in fact, straight lines for a considerable proportion of their length, deviations in the latter stages being due to variations in either $k$, $H_T$, or $k/\rho cp$. The deviation of the limestone curve from a straight line is due to the fact that decomposition is occurring over this range.

The straight-line portions of the curves for the three ores have slopes which are not greatly dissimilar, showing that the heating characteristics ($k$, $H_T$, and $k/\rho cp$) do not vary greatly for these ores.

Comparatively little is known of the thermal properties of iron oxides; Ralston[6] quotes some figures for the thermal conductivity of powdered $Fe_2O_3$ and some very doubtful figures for rods.

### Moisture and $CO_2$ Content

Four ores, Northamptonshire, Oxfordshire, Sproxton, and Frodingham, show effects which may be associated with the elimination of water.

The heating-rate curve of Northamptonshire (Figs. 1*c* and *d*) is very interesting. There is an initial retardation in the rate just above 100° C., due to the elimination of loosely held moisture, followed by a very marked further retardation at 300° C., due presumably to the elimination of water from hydrated ferric oxide (limonite). The thermal effect of the decomposition of any siderite present would not be detected as this would break down during the prolonged heating at 500–550° C., but the fact that this ore has the most rapid heating rate in the 600–850° C. region may be due to the evolution of heat by the oxidation of FeO from the decomposed siderite.

The heating curves for Oxfordshire ore (Figs. 1*c* and 2*a*) show that here the water is mainly loosely held, being eliminated between 100° and 150° C. There is, however, a certain amount of water of hydration present, as the curve (Fig. 1*c*) shows a further retardation in the heating rate in the 300–350° C. region.

The heating curves for Sproxton (Figs. 2*a* and *b*) suggest that both water of hydration and loosely held water may be present in different samples of this ore (*cf.* curves 9*A*, 9*B*, and 9*C*, Fig. 2*b*, and curve 9, Fig. 2*a*).

The heating curves for Frodingham (Fig. 3) show that most of the water is loosely held and is driven off between 100° and 150° C.

The heating curves of limestone (Figs. 1*c*, 1*d*, and 2*a*) show that, whilst the heating rate of limestone in the lower-temperature range is rapid as compared with ores, it is retarded considerably in the higher-temperature range, particularly above 600° C., owing to the decomposition of calcium carbonate.

If the effects of moisture and $CO_2$ elimination upon the transfer of heat to the centre of the lump were considered apart from other factors involved, calcination of the burden before charging would clearly be beneficial. These home ores, however, are in most cases readily broken down by the effect of heat and

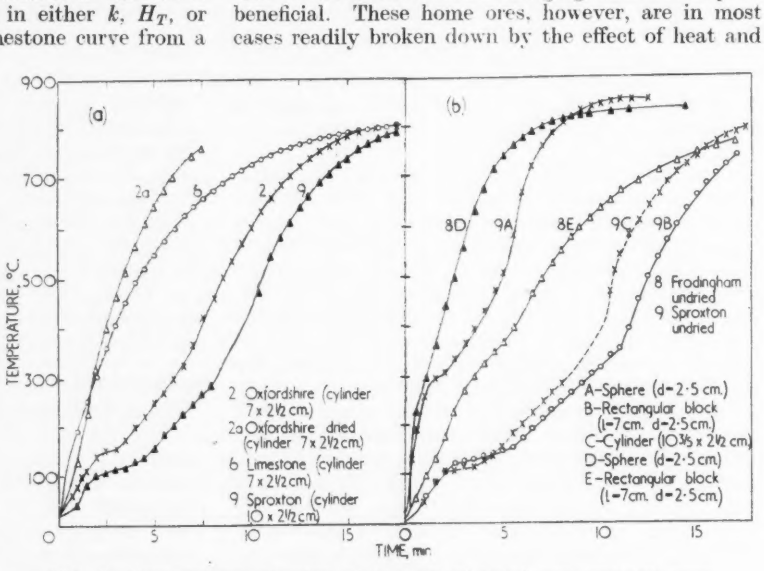



**Fig. 2—Comparison of rates of heating of dried and undried ores (room temperature to 870° C.)**

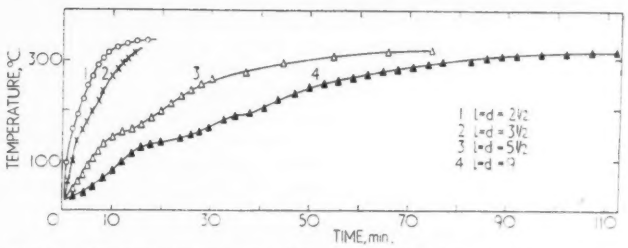



**Fig. 3—Effect of size on rate of heating undried Frodingham ore (room temperature to 350° C.)**

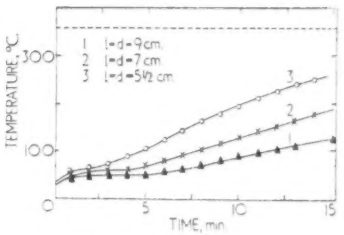



**Fig. 5—Effect of size on rate of heating. Cylinders of dried Frodingham ore (room temperature to 360° C.)**

carbon deposition, thus mitigating the effect of water and $CO_2$ elimination upon the rate of heating.

**Shape**

Specimens of various shapes were cut from two ores, Sproxton and Frodingham, whose heating rates represent the extremes amongst the ores studied (Grangesberg being neglected owing to the difficulties of machining); the diameter, in the case of spheres and cylinders, and the block thickness, in the case of cubes and rectangular blocks, being the same in all cases. This represents a simplified picture of the type of shape distribution which is found in practice in material retained between two screens, since one cross-section of the lumps will be limited by the screen sizes whilst the third dimension may vary considerably. Here the diameter or block thickness ($d$) remains the same, but the mass varies. If the mass is the same, $d$ will vary, being a maximum for a sphere and decreasing to a very small value for a long cylinder or block. Thus, the rate of rise of temperature at the centre of such a lump will be much greater than that for a sphere.

Assuming values for the thermal constants, the approximate centre temperatures of such shapes, *i.e.*, a sphere of diameter $d$ and a cylinder of the same mass and length $5d$, after the same times in the hot zone (900° C.) may be obtained from standard diagrams. The values thus obtained for two arbitrarily chosen times, $\theta$ and $\theta'$, are shown below :

Time $\theta$ : sphere, centre temp. approx. 400° C.
cylinder, centre temp. approx. 800° C.

Time $\theta'$ : sphere, centre temp. approx. 800° C.
cylinder, centre temp. approx. 895° C.

Whilst these figures have no absolute meaning they indicate the large difference in rate of rise of centre temperature between the sphere and the cylinder.

Figures 2*b*, 4*a*, and 4*b* show that in all cases where the diameter (or block thickness) is the same the shape has an important effect on the rate of heating of the specimen, particularly over parts of the heating period. For example, after 3-min. heating, the temperature at the centre of a sphere of dry Sproxton is some 350° C. higher than that at the centre of a rectangular block (Fig. 4*b*), the corresponding figure for dry Frodingham after a similar heating period being about 320° C. (Fig. 4*a*). With undried ores the effect is even larger, the difference in centre temperatures of a sphere and rectangular block of undried Sproxton after 8 min. being some 560° C. (Fig. 2*b*).

Figure 6 shows the rectified rate-of-heating curves

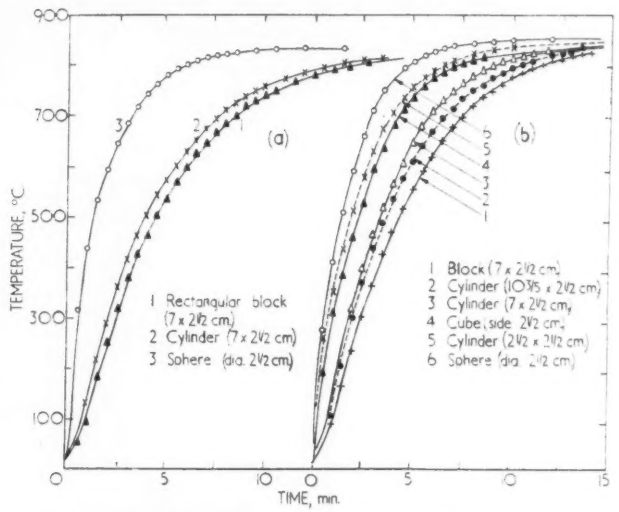



**Fig. 4—Rate of heating of various shapes of dried ore : (*a*) Frodingham, (*b*) Sproxton. Room temperature to 850° C.)**

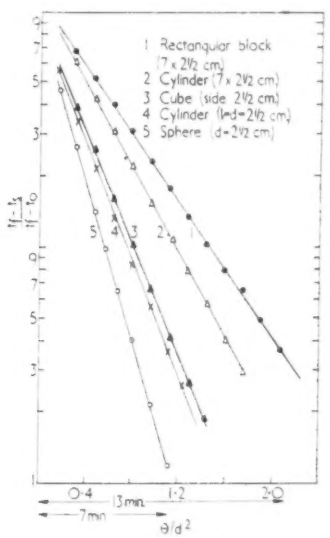



**Fig. 6—Influence of shape factor. Sproxton ore**

obtained from the data plotted in Fig. 4*b*. In this case all the thermal factors are the same, as are the diameters (or the equivalent measurement, the block thickness), and thus the only variable is the shape. The straight lines obtained have relative slopes which are in excellent agreement with those obtained by previous workers.[7]

**Size**

Four specimens were prepared from the same lump of undried Frodingham ore in the form of cylinders with the diameter equal to the length, with the following dimensions: $l = d = 2{\cdot}5$, $3{\cdot}5$, $5{\cdot}5$, and 9 cm., respectively. Figure 3 shows the rate of heating of these lumps from room temperature to 350° C., and the very marked difference in the rate of rise of the centre temperature can be seen; *e.g.*, the time taken for the centre of the specimen to approach to within 50° C. of the furnace temperature increases twelvefold as the diameter increases from $2\frac{1}{2}$ to 9 cm., which may be compared with the increase of 13 times in the diameter squared.

Similar rate-of-heating curves for cylinders of dried Frodingham ore with dimensions $l = d = 5\frac{1}{2}$, 7, and 9 cm., are shown in Fig. 5. The relationship between the times for the centre temperature to reach a given value and the diameter squared is similar, *e.g.*, the ratio of the times to reach 130° C. is $2{\cdot}5$, and the ratio of $d^2$ is $2{\cdot}7$.

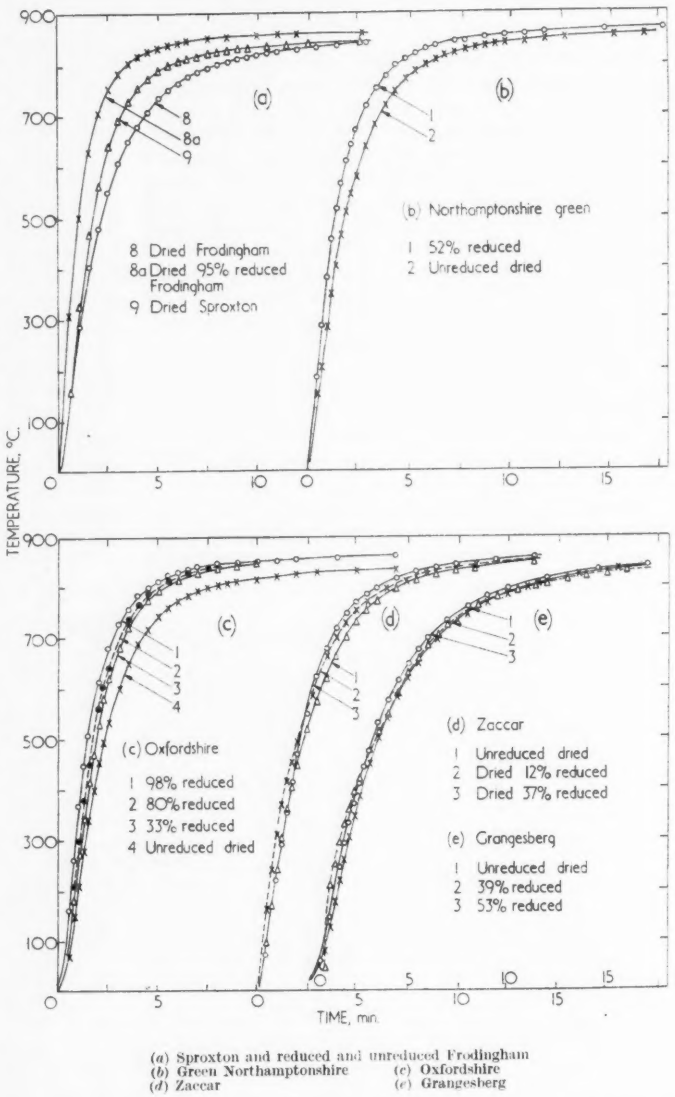
**Degree of Reduction**

It is important to know the effect upon the heating rate of the elimination of oxygen from the ore. The increase in metallic iron content, which might be expected to lead to an increase in thermal conductivity, is accompanied by a change to a more open structure which would have an opposite effect, and this, together with changes in the coefficient of heat transfer and the thermal diffusivity, makes it impossible to predict the behaviour of ores on reduction.

Thus, specimens of five ores, Frodingham, Northamptonshire, Oxfordshire, Zaccar, and Grangesberg, were prepared in the form of cylinders 4 cm. long × 2 cm. dia.; these were first reduced by hydrogen to the desired degree, as measured by the percentage of oxygen removed from the iron-bearing material between the limits $Fe_2O_3$ (0% reduced) and Fe (100% reduced), and then subjected to the normal heating treatment, except that this was now carried out in an atmosphere of nitrogen. Figure 7 shows that reduction of the ores causes, in general, an increase in the overall rate of heat transfer to the centre. The increase is not great and varies from ore to ore, but it should be noted that the specimens are small. Comparison with Fig. 1*f*, which shows the curves obtained with similar-sized specimens of various ores, shows that the difference between an unreduced and completely reduced ore is of the same order as that between different ores. Such change in heating rate will obviously be magnified with larger lumps of ore and is beneficial for furnace operation.

## SUMMARY OF RESULTS OF PRELIMINARY SURVEY

Neither the nature of the ore nor the degree of




(*a*) Sproxton and reduced and unreduced Frodingham
(*b*) Green Northamptonshire (*c*) Oxfordshire
(*d*) Zaccar (*e*) Grangesberg

**Fig. 7—Rate of heating of various reduced and unreduced ores (room temperature to 870° C.). Cylinder $l = 4$, $d = 2$ cm.**

reduction has a large influence on the rate of heat transfer to the centre of the lump. The shape of the specimen has a larger effect and its size has a much more marked influence. Moisture and $CO_2$ elimination cause a marked retarding of the rate of heat flow to the centre, but this effect may also cause fissuring and breakdown of the lumps, which would have the converse effect.

## HEAT TRANSFER WITH BLAST-FURNACE RATE OF HEATING

For the preliminary work the temperature of the surroundings of the ore was constant within certain limits, and the temperature at the centre was studied. In practice, the temperature of the surroundings of the ore increases as it descends in the furnace. The rate of increase will be more or less regular according to the regularity of furnace driving, but 160° C./hr. has been taken as an average for normal working. For this reason the behaviour of ore specimens heated at this rate between room temperature and 900° C. has been studied.

### Modification of Experimental Procedure

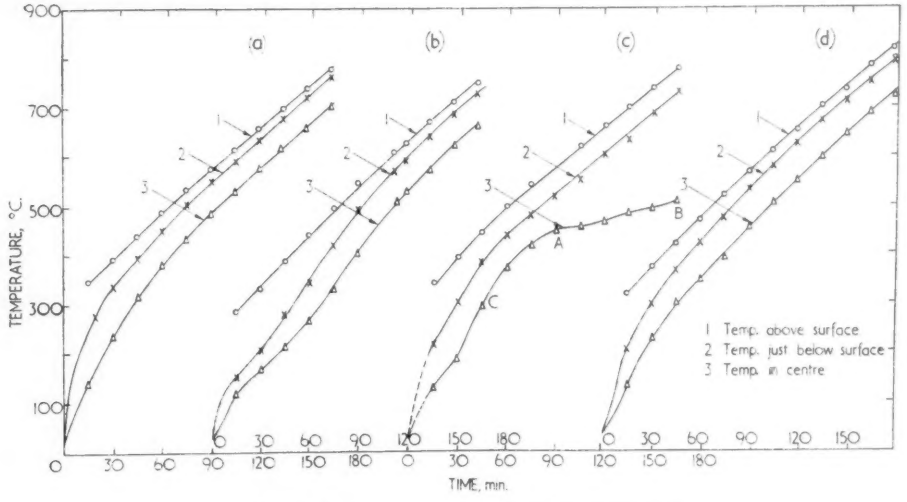
The desired linear rate of heating of 160° C./hr. for the furnace was secured by including in the furnace circuit an external resistance which was reduced stepwise by means of a manually operated multi-point control in a manner determined by blank experiments. The shape of the specimen was standardized as a cylinder of length equal to diameter. As before, a thermocouple was introduced into the centre of the specimen and for this work a second was now placed just below the surface at the middle of the cylinder. The temperature of the furnace and the two ore temperatures were recorded continuously during the experiment.

### Nature of Ore

Eaton, Northamptonshire, and Frodingham ores were investigated, the first being used dried and undried, the second undried, and the third dried. All samples were cylindrical, with $l = d = 9$ cm. The temperature/time curves are shown in Fig. 8. In each case, the linear relationship between temperature of surroundings and time was maintained, the slope of the temperature/time curve being 160° C./hr. With the dried ores, neglecting the early part of the experiment where the temperature difference between the ore and its surroundings is rapidly altering, both the surface and centre temperatures have a similar linear relationship with time and substantially the same slope (Figs. 8*a* and *d*).

The temperature lag between the surface of the ore and its surroundings is very similar for the two dried ores, being between 20° and 30° C. in each case. With undried Eaton, the lag at lower temperatures is greater, owing to the elimination of water over this range, but above 500° C. it is approximately the same. With Northamptonshire ore, however, the lag is considerably larger over the whole range, *i.e.*, some 50° C., probably because endothermic reactions occur throughout the experiment.

The temperature lag between the surface and the centre for dried and undried Eaton, after water elimination from the undried ore is complete, is 50–60° C., and for dried Frodingham approximately 75° C. Northamptonshire ore behaves very differently; after elimination of loosely held water, the normal parallel linear relationship is established (point *C*, Fig. 8*c*), and is maintained until the centre temperature reaches 420° C., when strong endothermic reactions, as for instance the decomposition of siderite and complex silicates, cause a very marked retardation of the temperature rise at the centre, completely




(*a*) Dried Eaton
(*b*) Undried Eaton
(*c*) Undried Northamptonshire
(*d*) Dried Frodingham

**Fig. 8—Time/temperature curves for various dried and undried ores. Rate of heating of furnace 160° C./hr. Cylinder $l = d = 9$ cm.**

destroying the normal relationship over the range *AB*, Fig. 8*c*.

**Size of Ore**

Besides the 9-cm. cylinder of dry Frodingham ore, two other cylinders, sized 7 and 5½ cm., were heated in the controlled manner, the curves obtained being shown in Fig. 9. The temperature lags between surface and surroundings and between centre and surface both increase as the size of specimen increases from 5½ to 9 cm., the former from 16° to 30° C., and the latter from 25° to 75° C. Figure 10 shows the overall temperature lag between the surroundings and the centre of the lump plotted against lump size. Point *F* is derived from an isolated experiment using hematite, and its exact position, and hence the precise course of the region *EF* of the curve, is open to doubt. It does, however, give an indication of the probable extent of the temperature lag in large lumps. From this graph the temperature of the centre of a lump of such an ore of any given size can be predicted for any furnace temperature ; *e.g.*, the centre of an 8-cm. (3¼-in.) lump in the 700° C. region is 630° C. With larger lumps the lag will be much greater with a correspondingly large increase in the extent to which thermally controlled reaction might be delayed ; *e.g.*, a 14-cm. (5½-in.) lump, assuming that reduction commenced at 400° C., would need to be at the 700–750° C. zone of the furnace before the temperature necessary for reduction had been reached at the centre, making no allowance for the alteration of thermal properties during descent. It should be noted, however, that this curve was derived mainly from experiments with dried Frodingham ore, and, strictly speaking, applies only to that ore.

## CHEMICAL REDUCTION

While the ore lumps are descending the blast-furnace and being heated, chemical processes are taking place simultaneously. Mention has already been made of the effect of water and $CO_2$ elimination and of the degree of reduction upon the temperature within the ore lump during heating ; but this temperature is not the only factor limiting the penetration of reduction into the lump. Chemical reaction rate and factors involving diffusion of the reducing gases to the surface of, and into, the lump and diffusion of the gaseous products of reaction from the lump are also involved.

Whilst much work has been carried out in the past on the reduction of iron ores (a full bibliography is given by Specht and Zapffe[8]), a large proportion of this has been concerned with the reduction of beds of small particles. Where the reduction of lump ores has been studied the investigators have in many cases been primarily interested in the more theoretical aspects and have thus used hydrogen as the reducing gas to avoid the complication of the reaction $2CO \rightleftharpoons CO_2 + C$.[3, 4, 9–13] Of these investigations the work of Kalina and Joseph[12] is of most direct interest, since they investigated the oxygen gradient into cylinders of ore of various types. The reduction of lump ores with CO or blast-furnace gas of various compositions has been carried out by a number of workers.[11, 14–16] In the work of Diepschlag, Zillgen, and Poetter,[14] the reduction was effected in stages at increasing temperatures, the gas composition varying at each stage to correspond with blast-furnace practice ; lumps of about 1 in. were used and the amounts of oxygen removed from ores of various types were compared and shown to be directly related to the porosity. Herzog,[16] using blast-furnace gas ($CO + N_2$) investigated the reduction of various ores and sinters with the temperature rising to 900° C. in 8 hr., but the size of the lumps used was rather small (approximately ½ in.). Tests were carried out with larger lumps at 900° C. and the extent of oxygen removal at the surface and centre was estimated.

Although this work throws considerable light on the problem of ore reduction, the extent of the penetration of reduction into various ores of widely different types

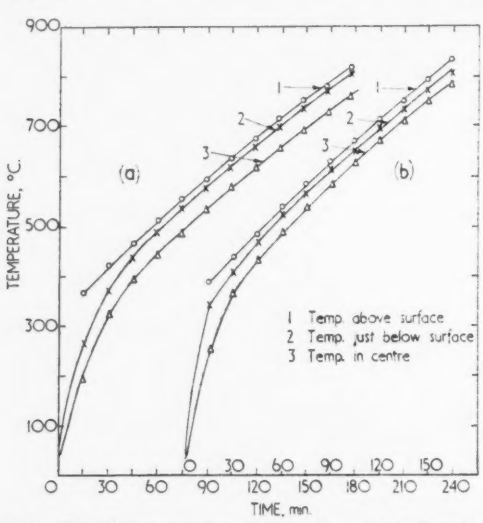



**Fig. 9—Time/temperature curves for dried Frodingham. Rate of heating of furnace 160° C./hr. (*a*) Cylinder *l* = *d* = 7 cm. (*b*) Cylinder *l* = *d* = 5½ cm.**

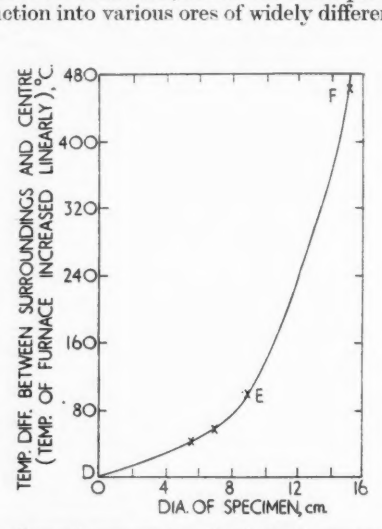



**Fig. 10—Overall temperature lag between surroundings and centre of specimen plotted against size of specimen**

under blast-furnace conditions still needs to be found, and thus it was decided to investigate the gradient of oxygen removal from the surface to the centre of the lump over a wide range of ores.

## EXPERIMENTAL PROCEDURE

The rate of heating of the lump was normally maintained at 160° C./hr., as in the later experiments on heat transfer into the lump. The heating was commenced at 300° C. and maintained at the standard rate for 4 hr. to a final temperature of 940° C.

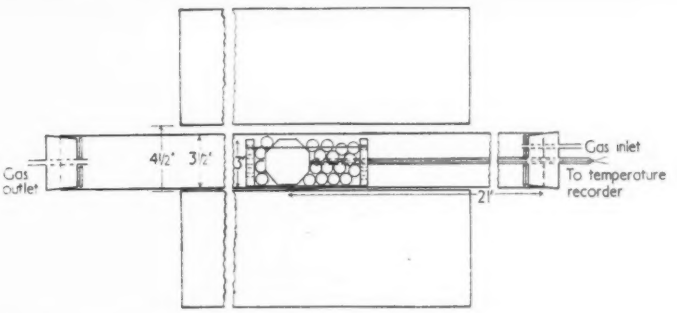



**Fig. 11—Section through furnace, showing position of lump in container**

Previous investigators had suggested that diffusion into or out of the lump was probably the most important factor involved in the process of reduction[11, 17, 18]; if this is true, or if the critical factor is the surface available for gaseous attack, the most important physical property will be the permeability of the ore which is closely linked with its porosity.[19] That porosity is the property of the ore that determines the reducibility was asserted by Diepschlag, Zillgen, and Poetter,[14] and the work of Joseph,[3] Lewis,[4] Kalina and Joseph,[12] and Saunders and Tress,[20] is in agreement with this assertion. The work of Philbrook[13] produced some anomalous results, but these he explained as being the result of the greater tendency of the less permeable ores to crack and split. Joseph,[3] from his porosity and reducibility results and certain observations of blast-furnace operation, constructed a graph relating optimum ore size for the blast-furnace with porosity.

Hence, for the ensuing work, the porosity of the ore was used to determine the size of the lump employed so that the penetration of chemical reduction should bear the same relationship to size in each case. The upper size limit for the most porous ore was imposed by the furnace capacity, and the sizes of the other ores were determined from the porosity by the use of a curve of the form employed by Joseph,[3] the porosities in each case being determined by the S.K. method.[20] The following table shows these values and the lump sizes of the various ores used :

| Ore | Porosity, % | Size, in. |
|---|---|---|
| Oxford Limy | 52·0 | 3¼ |
| Frodingham Silicious | 45·5 | 3¼ |
| Frodingham Limy | 43·5 | 3¼ |
| Sierra Leone | 43·0 | 3 |
| Northamptonshire Green | 42·2 | 3 |
| Djerissa | 40·9 | (3) 2¼ |
| Ouenza | 38·8 | 2¾ |
| Keloucha | 30·0 | (2½) 1⅝ |
| Konakry | 28·8 | 2¼ |
| Cleveland | 25·9 | 2¼ |
| May-sur-Orne | 18·2 | 1⅝ |
| Cumberland Hematite | 17·5 | 1⅝ |
| Oued Zem | 14·7 | 1⅝ |
| Rif | 12·5 | 1½ |
| Wabana | 6·7 | 1¼ |
| Grangesberg | 0·8 | ¾ |
| Gellivare | 0·8 | ¾ |
| Kiruna | 0·4 | ¾ |

In the case of Djerissa and Keloucha the figures in brackets are the sizes which should, on a porosity basis, have been used, but the specimens actually employed were the largest that could be cut from the lumps available.

The ideal shape for ore lumps would be spherical, when the oxygen gradient into the ore would be the same over the whole surface, or failing this, cylindrical, with the length large compared with the diameter, although this would have the disadvantage of needing a long constant-temperature zone within the furnace. Any angular shape will be less satisfactory as the oxygen gradient will not be the same at different points around the periphery. Neither spheres nor cylinders can, however, be cut from the harder ores without great difficulty and hence a near-spherical shape was adopted. This is a symmetrical 18-sided figure derived from a cube by removing each of the eight edges from two opposite faces in turn and finally the remaining four. Cutting was accomplished by means of a diamond wheel and, if carefully carried out, no signs of surface glazing were produced.

A single lump of the ore, cut to the desired size, was placed in a container (shown in position in the furnace in Fig. 11). The ends of the container were of refractory brick, perforated with ¼-in. holes to allow gas passage, and the sides and base were of sheet mica cemented to the refractory ends. The lump was placed about two-thirds of the way along the container, the remainder of which was packed with iron-free refractory spheres to keep the lump in position and to ensure final preheating of the gas. The Pt/13% Pt–Rh thermocouple in contact with the surface of the lump was sheathed with a thin coating of alumina cement, and the leads from it were led through a thin silica tube passing through the rubber bung, which was protected on the inside by an asbestos radiation shield, closing the upstream end of the furnace tube. This whole assembly was inserted into the furnace tube, which had been stabilized at 350° C., to a position such that the thermocouple was inside the constant-temperature zone of the furnace, and a slow stream of nitrogen was passed through. Adequate time (1 hr.) was allowed for the lump to reach the furnace temperature, which was now stabilized at 300° C. and, at the end of this time, blast-furnace gas of composition $2N_2 + CO$ was passed over the specimen at the rate of 330 litres/hr. The component gases were obtained from cylinders, passed through flowmeters and, after mixing, through a preheating furnace packed with broken silica to the reduction furnace. Simultaneously with the intro-

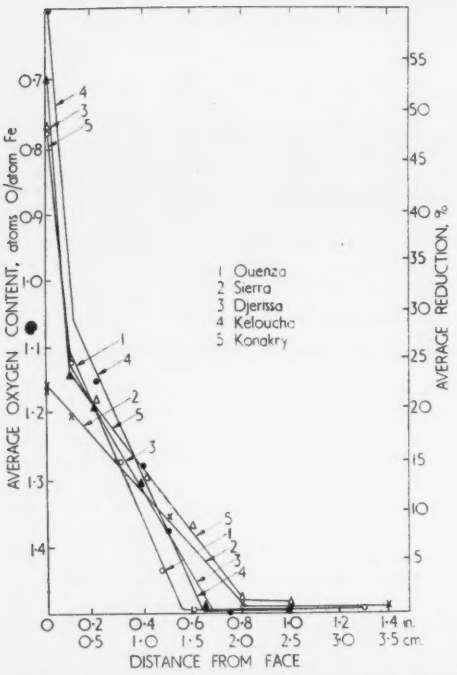



**Fig. 12—Penetration into iron-rich hematites of high permeability**

duction of the reducing gas, the controlled heating of the specimen was commenced. No attempt was made to follow the reduction by the estimation of the $CO_2$ produced, since carbon deposition over the lower temperature range and carbon solution at higher temperatures would render the results valueless. After 4 hr., when the temperature had reached 940° C., the flow of CO was stopped, the furnace was switched-off, and the specimen was cooled in a slow stream of $N_2$.

The linear rate of gas flow over the ore varied according to the area of free space around the lump, which was proportional to the size of the lump, and the packing of the refractory spheres around it. The upper limiting rate of flow was reached with a $3\frac{1}{4}$-in. ($8\frac{1}{4}$-cm.) lump and, whilst the smallest lump was $\frac{3}{4}$ in. (1·9 cm.), the packing of the refractory spheres around it reduced the free space to an estimated two-thirds of the tube cross-section. In addition, the rate of gas flow increased as the experiment progressed, owing to increased thermal expansion of the gas. Because of these factors no single figure can be given for the linear rate of flow of gas over the ore, but probable limits for the range of flows are :

| | Rate of Flow of Gas : At 300° C. cm./sec. | At 940° C. cm./sec. |
|---|---|---|
| Small-sized ore lump | 4·5 | 9·6 |
| Maximum lump size ($3\frac{1}{4}$ in. = $8\frac{1}{4}$ cm.) | 18·4 | 39 |

Thus the ratio between maximum and minimum gas flows at any given temperature is of the order of 4/1 for extreme lump sizes.

Direct comparison of the effect of different flows over different-sized lumps of similar material is possible for only one ore (Ouenza), and here the difference in rates of linear flow between the largest ($2\frac{3}{4}$-in.) lump and the smaller lumps is only 2 : 1. Any increase in the degree of reduction of the ore due to increased rate of flow is more than counter-balanced by other effects. In fact, the degree of surface reduction and the oxygen gradients within the lump do not seem to be linked in any way with changes in the flow rate within the limits used in this investigation.

Difficulties in preheating larger volumes of gas precluded the use of flows greatly in excess of that used for this work, and direct comparison of the extent of surface reduction and depth of penetration with different gas flows by using large and small lumps is not possible owing to the influence of other factors.

After removal from the furnace the specimen was examined and, if seriously cracked, was mounted in a thermo-setting plastic by surrounding it with this material and heating to 100–120° C. to bring about polymerization. The specimen was then cut along the axis of the gas flow for examination. The nature and extent of any colour changes across the section and any other physical facts were noted. A series of samples was obtained either, in the case of the softer ores, by making a series of concentric shallow borings from the surface to the centre across the section, or, in the case of the hard ores, by obtaining a thin (2–3-mm.) section across the specimen by a second cut parallel to the first, and breaking this so as to obtain a similar series of samples. The oxygen gradient from the surface to the centre of the lump was obtained by chemical analysis of these samples for Fe, Fe″, and Fe‴.[21]

The samples from the surface of the lump consist of flakes or chips of material with a thickness of less than 1 mm., taken from points around the periphery at the line of sectioning ; hence they represent a mean value. In some specimens the oxygen gradient was very steep near the surface and thus a small variation in the average thickness of the specimen may have a large effect upon the degree of reduction, *e.g.*, repeat analyses on different samples of the surface of the reduced Sierra Leone gave values of 22·5 and 23·1% for the degree of reduction, whereas repeat analyses of the surface of the 1-in. Ouenza specimens, where the oxygen gradient is much steeper, gave values of 82·5 and 88·1%. The variation in the free-iron content over the surface of hard ores is discussed later.

## RESULTS

The behaviour of the ores studied under the standard reducing conditions enables them to be divided into four groups, the ores in each group behaving in general in a similar manner. The groups are as follows :

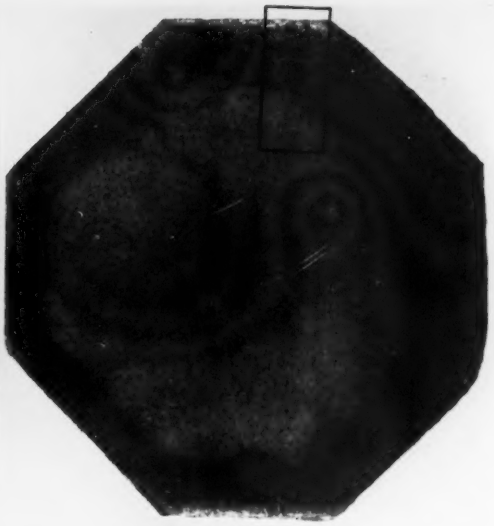
(1) Iron-rich hematites of high permeability : Ouenza, Sierra Leone, Djerissa, Kelouchа, and Konakry

(2) Iron-rich hematites of low permeability : Maysur-Orne,* Cumberland Hematite, Oued Zem, Rif, and Wabana*

(3) Magnetites : Grangesberg, Gellivare, and Kiruna

* These ores are not true hematites, but are placed in this group for convenience.

(a)



(a)

(b)

(b)

**Fig. 13—(*a*) Section through reduced lump of Sierra Leone ore. (*b*) Enlargement of the area outlined in (*a*)**

**Fig. 14—(*a*) Section through reduced lump of Oued Zem ore. (*b*) Enlargement of the area outlined in (*a*)**

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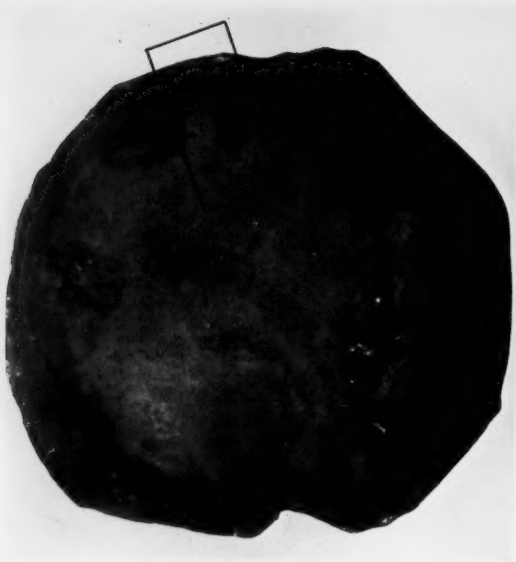
(a)



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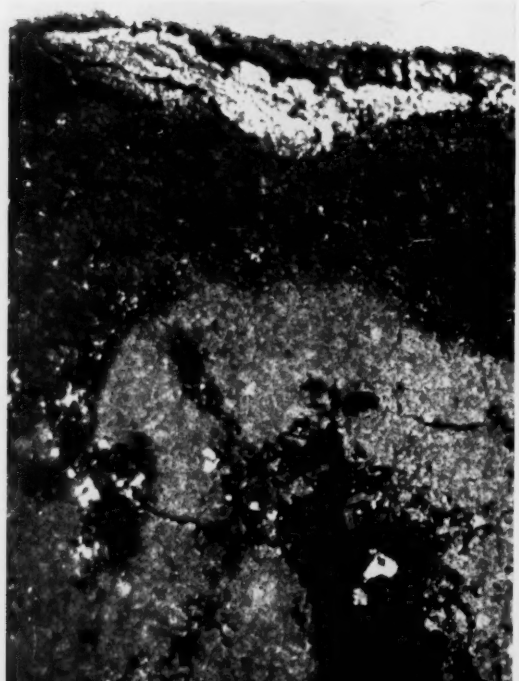

Fig. 15—(*a*) Section through reduced lump of Rif ore. (*b*) Enlargement of the area outlined in (*a*)

(a)

(b)

Fig. 16—(*a*) Section through reduced lump of silicous Frodingham ore. (*b*) Enlargement of the area outlined in (*a*)

*Wild and Saunders*]
*To face p.* 207]

(4) Permeable home ores : Oxford Limy, Frodingham Limy, Silicious, Northamptonshire Green, and Cleveland.

For each ore a curve was drawn showing the oxygen content, expressed as atoms of oxygen per atom of iron and also as percentage reduction, at any annular position, plotted against the position of this annulus relative to the surface of the lump. Each point represents an average figure as the penetration is not regular, particularly with some ores, around the lump.

**(1) Iron-Rich Hematites of High Permeability** (Fig. 12)

The reduced Ouenza was crumbly over parts of the surface and small flakes could readily be detached, but there were no large cracks or fissures. When sectioned, a sharply defined colour change between the unchanged ore and the reduced outer zone was visible, this boundary being circular and concentric with the lump. The oxygen gradient is shown in curve 1 of Fig. 12, and consists of three sections, a rapid fall in the degree of reduction over the surface layer, an intermediate portion with a much less rapid fall, and the central unreduced zone.

After reduction the specimen of Sierra Leone was appreciably fissured. Sectioning showed a definite colour change between the outer reduced portion and the unreduced core, but this was neither so regular nor so sharply defined as in the case of Ouenza. Figure 13*a* is a photograph of a section across the reduced lump in which the extent of the reduced annulus is plainly visible, and Fig. 13*b* is an enlargement of the portion outlined in Fig. 13*a*, showing the transition from the unreduced core, where the individual grains of hematite can clearly be seen, to the outer reduced zone. The fact that reduction does not penetrate more deeply along the cracks suggests that they were formed during cooling. The oxygen gradient (curve 2, Fig. 12) is of a different type from that of Ouenza, being uniform from the surface to the unreduced core, the degree of reduction at the surface being much less than in the case of Ouenza. Whilst Sierra Leone is the only ore in this group to show this type of oxygen gradient, most of the permeable home ores (group 4) have oxygen gradients of this form (*see* Fig. 19).

The reduced specimen of Djerissa closely resembled that of Ouenza. Sectioning revealed a sharp, well-marked boundary between the reduced and unreduced portions, but the reduction zone was not so regular in depth as that of Ouenza. Over parts of the periphery a narrow band ( $<1$ mm.) of free iron was visible. Curve 3, Fig. 12, shows that the oxygen gradient from the surface to the centre is very similar to that of Ouenza.

After reduction the lump of Keloucha resembled the reduced specimen of Ouenza, both outwardly and in section, except that, as with Djerissa, a narrow free-iron zone was visible over part of the periphery. The oxygen gradient (curve 4, Fig. 12) is similar to that of Ouenza.

Konakry ore was very irregular in structure, containing large fissures and pockets of softer ore, but as typical a piece as possible was taken for reduction. After reduction the surface was cracked and tended to flake off. Deposited carbon was present over parts of the surface, but not as a continuous film over

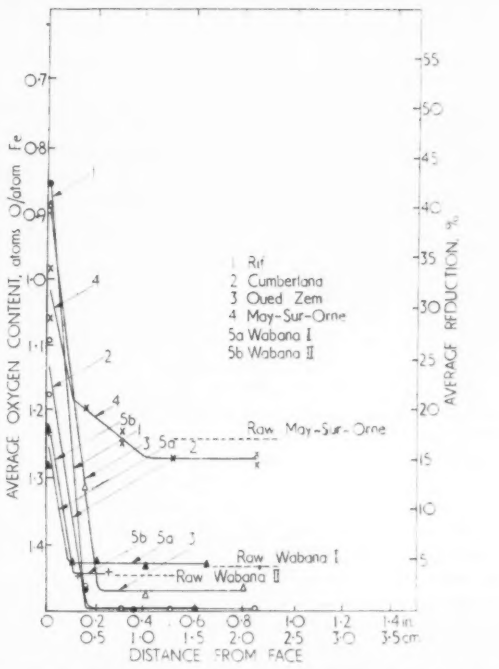


**Fig. 17—Penetration into iron-rich hematites of low permeability**

the whole lump. Sectioning revealed a very irregular unreduced core at the centre and a narrow zone of free iron over parts of the periphery. Samples for analysis were taken from a sector of the face where the penetration appeared most uniform. Curve 5, Fig. 12, shows that the oxygen gradient is of the usual type.

**(2) Iron-Rich Hematites of Low Permeability** (Fig. 17)

May-sur-Orne is not a true hematite, but it is most convenient to consider it under this heading. The sample used contained 22·7% Fe″ and 22% Fe‴; about 12% of the former was present as siderite, the remainder presumably being combined with the alumina (11·1%) and silica (10·6%).

The reduced ore was hard and, when cut, showed no colour change. Curve 4 of Fig. 17 shows the oxygen gradient from the surface to the centre ; it is similar to those of most of the permeable ores (Fig. 12), consisting of an initial steep fall in the degree of reduction, followed by a slower fall to the unreduced core. The degree of reduction of the raw May-sur-Orne is also shown.

The reduced lump of Cumberland hematite was somewhat cracked, but with no signs of disintegration. Small areas of deposited carbon were present on the surface. On sectioning, no colour change was visible. Curve 2, Fig. 17, shows the oxygen gradient from the surface inwards ; this is steep, with reduction penetrating only a thin outer layer of the lump.

On reduction, the surface of the Oued Zem lump was soft, with a marked tendency to flake off. Sectioning revealed a well-marked boundary between reduced

and unreduced zones, the reduced zone being wider on one side where there was a narrow visible fissure across the section. In Fig. 14*a*, a photograph of a section through the reduced lump, the irregularity of the reduced zone and the sharp demarcation between it and the unreduced core can be clearly seen ; Fig. 14*b* is an enlarged photograph of the portion outlined in Fig. 14*a*. A narrow zone of free iron is visible at the surface ; it is variable in thickness round the section and is not continuous. The demarcation between the reduced zone and the unreduced core is not well defined at this magnification, owing to the nature of the matrix. Curve 3, Fig. 17, shows the oxygen gradient, which is similar to that of Cumberland hematite, with rather greater surface reduction and deeper penetration.

The surface of the reduced Rif specimen was very soft and flaky. Sectioning revealed a sharp boundary near the face of the ore, the thickness of the visibly reduced zone being somewhat irregular due to cracks and fissures in the lump. This is clearly shown in Fig. 15*a*, a photograph of a section through the reduced lump, where the way in which the surface tends to flake off and the manner in which the penetration depth is influenced by cracks and fissures is plainly visible ; Fig. 15*b* is an enlarged photograph of the area outlined in Fig. 15*a*. Here the reduced zone is well defined with a layer of free iron at the face ; the irregularity of the reduced zone and the manner in which the surface tends to split off is again clearly indicated. Curve 1 of Fig. 17 shows the oxygen gradient to be very steep and the extent of the penetration small.

Wabana contains a certain amount of ferrous iron, in addition to hematite. The reduced specimen of this ore, which had a considerable quantity of carbon deposited over the surface, was found to be in two pieces. The larger, which comprised some three-quarters of the original lump, was sectioned in the normal manner. No reduction boundary was visible. The oxygen gradient is shown in curve 5*a*, Fig. 17, and the penetration of the reduction is small.

The reduction of Wabana was repeated with another specimen from a fresh consignment. At the end of the reduction the lump was shattered, there being two large pieces and a large number of smaller ones, mostly thin flakes, decreasing in size to dust. Over parts of the face of the ore and mixed with the smaller lumps was a considerable quantity of deposited carbon. An examination of the material showed that disintegration started at a fairly early stage, since there was deposited carbon on some of the inside faces and the degree of reduction on these faces was very similar to that of the outside of the lump, whilst the nature of several deep cracks through the largest lump suggested that the break-up was continuing. Analysis of the small pieces and ore dust (separated magnetically from the carbon) showed these pieces to be very considerably reduced (59·7%). The percentage reduction of two different portions of the surface was found, firstly that directly below a deposit of carbon and secondly below a carbon-free region. In the first case the degree of reduction was 33·0% (free iron 13·0%), and in the second 18·1% (free iron 1·6%). This shows that, in this case at any rate, the free iron is not regularly distributed over the face of the reduced lump and, since free iron is a catalyst for the reaction $2CO \rightarrow CO_2 + C$, the carbon will be deposited preferentially over that part of the surface where there is most free iron. The irregularity of the free iron at the face may be due to the fact that reaction spreads from a nucleus of free iron rather than proceeding by the formation of a second nucleus at a spot remote from the first. This fact probably explains the non-reproducibility of analytical results for surface samples of other ores in this group, *e.g.*, two surface samples of Cumberland hematite have reduction percentages of 21·6 and 27·0, and similar figures for two samples of May-sur-Orne are 29·1 and 34·1%.

The oxygen gradient, obtained from the largest lump of the reduced ore, is shown in curve 5*b*, Fig. 17, taking the lower value for the surface reduction, and is very similar to that of the first Wabana specimen. The percentage reduction of the raw Wabana ores is also shown.

A lump of Wabana from the same consignment as the previous specimen was heated in air at the standard rate. Disruption commenced below 600° C. and continued over the remainder of the heating range. A second lump similarly heated in air disintegrated with considerable violence around 400–500° C.

Examination of samples of Wabana showed that certain of the bedding layers were relatively rich in clay minerals, some lumps being much more affected than others. Differential thermal analysis showed a marked endothermic reaction at 550° C., identifiable as the decomposition of a clay mineral ; chemical analysis showed that heating to 650° C. resulted in the evolution of 1·5–3·0% of $H_2O$ and about 0·5%

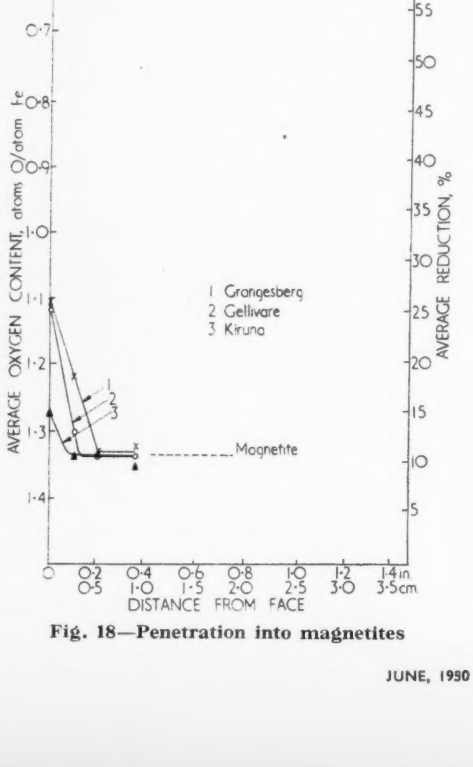


**Fig. 18—Penetration into magnetites**

of $CO_2$. The mechanism of the disintegration can thus be seen to be the decomposition of the clay minerals with evolution of water and $CO_2$, which cannot easily escape through the relatively impermeable matrix (porosity 6·7%) and thus splits the lump along the bedding layers to give flakes of ore.

**(3) Magnetites** (Fig. 18)

The particular lump of Grangesberg ore from which the specimen for reduction was cut was a very low-grade sample containing only some 40% of magnetite ; chemical analysis showed the remainder to be mainly silica (35·3%) and alumina (17·5%), together with smaller amounts of lime (3·4%) and magnesia (2·6%). Microscopical examination showed the specimen to consist essentially of a granular groundmass of quartz, with bands of biotite (mica) and apatite ; the magnetite crystals are small and are not usually in closely packed contact with one another. The reduced lump was very easily crumbled and a first attempt yielded no satisfactory samples for analysis as the whole lump crumbled to pieces when sectioning was attempted. A second specimen was set in plastic after reduction and, with careful handling, gave a complete section which was subsequently subdivided for analysis. With the magnetite lumps employed the small quantity of material from the centre of the section available for analysis resulted in the value for the oxygen content in this region being less accurate. Curve 1, Fig. 18, shows the oxygen gradient to be quite steep and the penetration of reduction small.

The sample of Gellivare was much richer in iron, containing some 85% of magnetite. Microscopical examination showed a coarsely granular aggregate of magnetite with some apatite and a mineral of the amphibole group (a complex silicate). The crystals are large and of a good shape and have a distinct segregation into parallel planes. The reduced lump was very crumbly at the surface and the same careful handling was necessary to obtain specimens for analysis. Curve 2, Fig. 18, shows that the oxygen gradient is similar to that obtained with Grangesberg, but somewhat steeper, and that the penetration of reduction is less.

The Kiruna used for reduction was very hard and dense and consisted of almost pure magnetite (98–100%). Microscopical examination showed the specimen to be very fine-grained and to have no major constituents other than magnetite. The crystals form an interlocking and interpenetrating framework which is responsible for the great toughness of the rock. The reduced sample was very hard and showed no tendency to crumble. Curve 3, Fig. 18, shows that Kiruna is very little reduced, even at the surface, and that only a very thin layer is penetrated by the reducing gases.

The differences in behaviour of the magnetites thus seems to be due to the opening up of the matrix by the presence of impurities which allows freer access for the reducing gases.

**(4) Permeable Home Ores** (Fig. 19)

The ores in this category are very variable in composition and it was difficult to cut a specimen which would have a reasonably uniform constitution throughout. In a number of cases, more than one specimen had to be reduced and sectioned before a satisfactory specimen was obtained. Care was taken in selecting a lump which appeared to have a uniform mineral distribution.

The sample of Oxfordshire Limy ore from which the specimen for reduction was cut, had the following analysis :

| | % | | % |
|---|---|---|---|
| Total moisture | 15·8 | Alumina | 12·1 |
| Calcium carbonate | 16·0 | Silica | 12·2 |
| Magnesium carbonate | 3·0 | Ferric oxide | 40·0 |
| | | Ferrous oxide | 0·3 |

On reduction it was immediately apparent that the lump, which was friable on the surface, had a variable iron content, and sectioning clearly revealed this. However, the iron content appeared fairly uniform over rather more than half the section, and samples for analysis were taken from this region ; it was just possible to discern on this area of the face the colour change associated with reduction. Curve 1, Fig. 19, shows the oxygen gradient from the surface to the centre of the lump ; the slope is uniform from the surface to the unreduced core, the degree of reduction is not high at the surface, but the penetration of reduction is considerable.

The sample of Frodingham Limy ore used for reduction had the following composition :

| | % | | % |
|---|---|---|---|
| Total moisture | 4·3 | Silica | 3·6 |
| Calcium carbonate | 54·4 | Alumina | 3·0 |
| Magnesium carbonate | 4·5 | Ferric oxide | 21·5 |
| | | Ferrous oxide | 5·5 |

After reduction the surface of the sample was friable

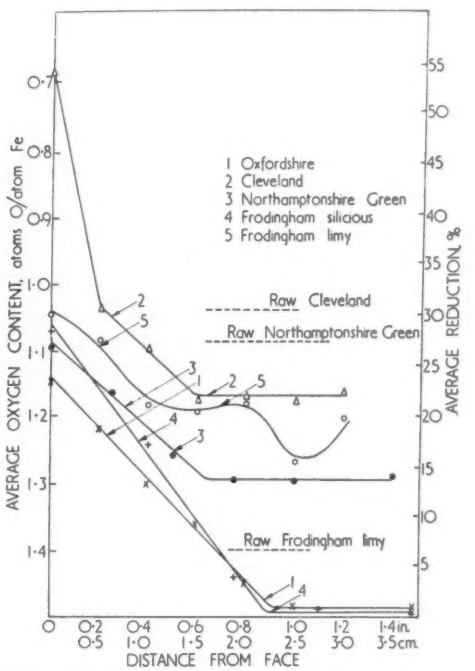


**Fig. 19—Penetration into permeable home ores**

and the variable iron content of the sample could be seen ; sectioning revealed no visible reduction boundaries. Curve 5, Fig. 19, shows that the oxygen gradient into the lump is less uniform than is normally the case. The degree of reduction of the raw ore is also shown and is considerably less than that of the centre of the ore, there being apparently no unreduced core. The reason for this is apparent from the chemical analysis of the ore which suggests that the matrix is calcite, in which are nodules of iron-bearing material (limonite and some siderite or chamosite). On heating, the calcite will decompose, with the evolution of $CO_2$, thus opening up the matrix for the entry of reducing gas. Since the iron-bearing material is present in isolated nodules it is possible for the reducing gas to penetrate deeply into the specimen without loss of reducing power, thus effecting reduction at points right to the centre of the lump. Since the distribution of iron through the lump is irregular, the degree of reduction at points away from the surface of the lump is not uniform.

The sample of silicious Frodingham ore had an analysis as follows :

| | % | | % |
|---|---|---|---|
| Total moisture | 13·0 | Silica | 18·0 |
| Calcium carbonate | 1·8 | Alumina | 17·2 |
| Magnesium carbonate | 2·5 | Ferric oxide | 47·5 |

On reduction the lump shrank appreciably (from 3·2 in. to 2·9 in. dia.) and was much harder in texture. The lump had cracks in it which were shown, on sectioning, to penetrate deeply. A regular circular boundary between the visible reduced and unreduced zones, however, suggested that these cracks had developed during the cooling. Figure 16*a* is a photograph of a section through the reduced lump in which the cracks developed during the experiment, and the well-marked reduced zones are clearly visible. Figure 16*b* is an enlarged photograph of the outlined area in Fig. 16*a*, where a discontinuous layer of free iron can be seen at the face ; examination of the section as a whole shows superficial zones of free iron of variable thickness and extent. Curve 4, Fig. 19, shows that the oxygen gradient into the lump is very similar to that of Oxfordshire ore (curve 1, Fig. 19).

The first sample of Northamptonshire green ore that was reduced consisted of two distinct portions, with a very sharp boundary ; the smaller portion was distinctly iron-rich, containing 50–55% of iron, whilst the larger portion was a lean ore with an analysis of about 25% of iron. A second sample of Northamptonshire green ore was thus cut and reduced and, whilst it was not entirely uniform, the larger portion had a uniform appearance, and from this samples were obtained for analysis. The analysis of this portion of the reduced lump was :

| | % | | % |
|---|---|---|---|
| Silica | 7·4 | Magnesia | 3·7 |
| Alumina | 18·0 | Iron oxides | 54·0 |
| Lime | 13·1 | | |

Curve 3, Fig. 19, shows the oxygen gradient into the centre of the lump ; this is the usual type for these ores, with a horizontal portion at 13·7%. The degree of reduction of the raw ore is also shown and has a very much higher reduction level (27·6%). For the centre core to have fallen to the lower reduction level, oxidation must have taken place at some time during the experiment. Whilst insufficient sweeping out with nitrogen might allow oxidation in the early stages of the experiment, it is difficult to see how this could affect the centre of the lump. During heating, $CO_2$ is liberated within the lump by the decomposition of siderite and later of calcite. This could effect oxidation of the FeO to $Fe_3O_4$, provided that its concentration was above the equilibrium value (50% of $CO_2$ at 550° C., 75% at 900° C.). Calculation of the $CO_2$ available from carbonate decomposition shows that this is adequate to effect oxidation to the level found at the centre of the lump under the existing conditions.

The analysis of the Cleveland ore used for reduction was as follows :

| | % | | % |
|---|---|---|---|
| Total moisture | 5·3 | Silica | 10·7 |
| Calcium carbonate | 6·6 | Alumina | 13·4 |
| Magnesium carbonate | 9·0 | Ferric oxide | 1·1 |
| Ferrous carbonate | 23·9 | Ferrous oxide* | 15·5 |

* Presumably combined with the alumina and silica.

During reduction a number of small pieces broke off the specimen ; subsequent analysis of the inside faces of these pieces established that they broke off during the heating as they were reduced to approximately the same extent as the outside face. When sectioned, the lump revealed no visible reduction boundary. Curve 2, Fig. 19, shows that the oxygen gradient into the lump is of the same form as that associated with permeable rich hematites (Fig. 12) ; the degree of reduction of the raw Cleveland is also shown and, as in the case of green Northamptonshire ore, oxidation has taken place within the lump. Similar calculations show that sufficient $CO_2$ is available to effect oxidation whilst maintaining the $CO/CO_2$ equilibrium. Oxidation of this type, but to a lesser extent, appears to take place in the specimen of May-sur-Orne (Fig. 17).

In a subsequent experiment a lump of Cleveland ore was heated in a stream of $N_2$ from 300° to 940° C. The water and $CO_2$ evolved were first absorbed and the remaining gases were then passed over heated copper oxide to oxidize any CO to $CO_2$. By this means, CO was shown to be present in the exit gases and could only have come from the oxidation of FeO to $Fe_3O_4$. After cooling in $N_2$ the lump was shown to be uniformly oxidized to approximately the same degree as the unreduced core of the previous lump (24·4% reduced, compared with 22·0%).

## VARIATION OF EXPERIMENTAL CONDITIONS

Besides studying the extent of the penetration of reduction, under the standard conditions, into these 18 ores, the effect upon the behaviour of Ouenza, taken as the reference ore, of the following modifications to these standard conditions was assessed :

Variation in the size of the lump
Varying heating rates
Effect of an initial overall partial reduction.

### Variation in Size of Lump

Specimens of Ouenza of size $2\frac{3}{4}$, 2, $1\frac{1}{2}$, and 1 in., were cut from the same lump of ore and reduced in the manner already described. After cooling, they were sectioned and the oxygen gradients from the surface

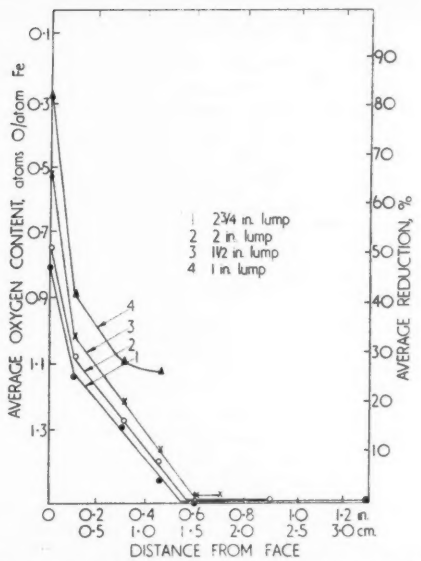


**Fig. 20—Penetration into Ouenza ore with varied lump size**

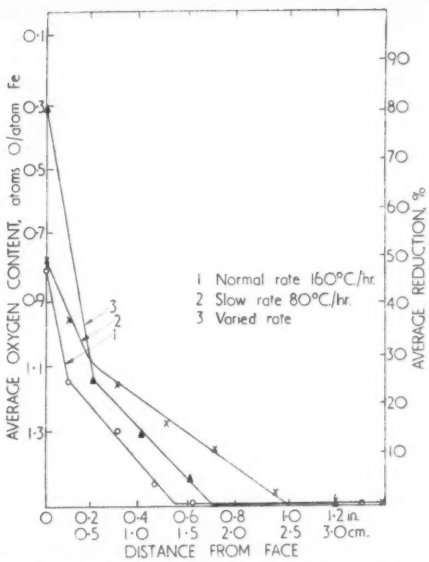


**Fig. 21—Penetration into Ouenza ore with varied heating rates**

to the centre of the lump were obtained ; these are shown in Fig. 20. The extent of penetration into the lump increases slightly as the size of the lump decreases, *i.e.*, from 0·55 to 0·60 in. as the lump size decreases from $2\frac{3}{4}$ to $1\frac{1}{2}$ in. The extent of the reduction within the reduced annulus, however, increases to an appreciable extent as the size of the lump falls (curves 1–3, Fig. 20). When the lump is of such a size that penetration can reach the centre, the extent of reduction in this region increases rapidly (curve 4, Fig. 20), presumably because the mass of material to be reduced falls rapidly.

The sizes of the ore lumps used for reduction were estimates, based on the porosity, of the sizes which would actually be suitable for use in practice, and thus corrections for the influence of size of specimen upon penetration are necessary only when deviations from this initial estimate are appreciable. Apart from the cases of Djerissa and Kelouchа, where smaller lumps than that indicated by the porosity were taken and where suitable small corrections have been made by comparing the extent of penetration with that obtained with a lump of Ouenza of the same size, the maximum deviation from the anticipated size occurs with Konakry, and here the correction to the penetration figure is very small and necessitates a correction of less than $\frac{1}{4}$ in. in the comparative size (*see* the table on p. 212).

### Variation of Heating Rates

Besides being reduced at the standard rate of 160° C./hr., a specimen of Ouenza (of size $3\frac{1}{4}$ in.) was reduced at half this heating rate (300° to 940° C. in 8 hr.). The specimen was cracked to a much greater extent than that heated at the normal rate, probably because of the greater length of time during which it was subjected to carbon deposition. Figure 21 shows that the oxygen gradient near the surface is less steep with the lower heating rate, but that the extent of penetration of the reduction was considerably greater (1·0 in., compared with 0·55 in.).

Another specimen of Ouenza (of size $2\frac{3}{4}$ in.) was heated over the normal temperature range for the standard time, but with a varied heating rate. During the first 70 min. the temperature was raised from 300° to 760° C. (heating rate 400° C./hr.), whilst for the remaining 170 min. the heating rate was 64° C./hr. The degree of reduction at the surface is very much greater and the extent of the reduction penetration is also appreciably greater (0·70, compared with 0·55 in.) (Fig. 21).

### Effect of Initial Uniform Partial Reduction

Two specimens of Ouenza ore were cut from the same lump and exposed to CO until the overall degree of reduction of the lumps had reached approximately 50%. Reduction was carried out by circulating pure CO over the ore, heating to 850° C. in a closed system and absorbing the $CO_2$, more CO being added from time to time to maintain atmospheric pressure. The $CO_2$ was weighed at intervals and the CO was pumped off when the desired weight of $CO_2$ had been evolved. Homogenization of the lumps was effected by the circulation of an equilibrium mixture of CO and $CO_2$ over them for a prolonged period (about 200 hr.). Drillings from different depths of one of the lumps were analysed and showed that the degree of reduction was substantially the same across the lump (58% at the surface, 53% at the centre).

The second lump was subjected to the standard heating and reduction treatment and was subsequently cooled and sectioned in the normal manner. This revealed a well-marked zone of free iron around the surface, with a rather diffuse boundary separating it from the central zone. Figure 22 shows the oxygen gradient into the reduced ore. For the outer layer

the degree of reduction is about 90% ; inside this there is a zone in which the oxygen gradient is steep, being of the same order as that in the surface layer of a normal lump, whilst inside this the gradient is much less steep. Penetration of the reducing gases reaches the centre, and the reduction in this region is similar to that near the centre of a 1-in. lump. During the latter stages of the reduction the reducing gases penetrate the outer layer with little or no loss in reduction potential, and thus the oxygen gradient beyond this layer is similar to that of a smaller lump where penetration reaches the centre (curve 4, Fig. 20). The overall penetration is greater with the reduced lump since inital reduction has made the structure of the ore more open.

## PENETRATION OF REDUCTION

From the point of view of this investigation the most important factor is the depth of penetration of the reducing gases. The value of this for any ore is given by the abscissa of the point at which the oxygen-content/distance-from-face curve flattens to a horizontal line. The values of this limit of penetration are shown in the following table, where the porosities are also given. The values in column (iv) are determined by a comparison of the penetration values for each ore with that of Ouenza and the adoption of a 3-in. lump of this ore as a standard.

| Ore (i) | Porosity, % (ii) | Depth of Penetration of Reducing Gases, in. (iii) | Comparative Size, in. (3-in. Ouenza as standard) (iv) |
|---|---|---|---|
| **Iron-rich hematites of high permeability** | | | |
| (1) Ouenza | 38·8 | 0·55 | 3 |
| (2) Sierra Leone | 43·0 | 0·8 | 4½ |
| (3) Djerissa | 40·9 | 0·68 | 3½* |
| (4) Keloucha | 30·0 | 0·62 | 3* |
| (5) Konakry | 28·8 | 0·8 | 4½ |
| **Iron-rich hematites of low permeability** | | | |
| (6) May-sur-Orne | 18·2 | 0·4 | 2¼ |
| (7) Cumberland | 17·5 | 0·17 | 1 |
| (8) Oued Zem | 14·7 | 0·22 | 1¼ |
| (9) Rif | 12·5 | 0·15 | ⅞ |
| (10) Wabana | 6·7 | <0·1 | < ½ |
| **Magnetites** | | | |
| (11) Grangesberg | 0·8 | 0·2 | 1 |
| (12) Gellivare | 0·8 | 0·13 | ¾ |
| (13) Kiruna | 0·4 | <0·1 | < ½ |
| **Permeable home ores** | | | |
| (14) Oxfordshire | 52·0 | 0·9 | 5 |
| (15) Frodingham Limy | 43·5 | ... | ... |
| (16) Frodingham Silicious | 45·5 | 0·87 | 4¾ |
| (17) Northamptonshire Green | 42·2 | 0·65 | 3½ |
| (18) Cleveland | 25·9 | 0·6 | 3¼ |

* To obtain the comparative size for column (iv) the penetration depths for Djerissa and Keloucha were compared with that for 2 in. and 1½ in. Ouenza, respectively.

In Fig. 23 the values of the penetration depth, shown in column (iii) above, have been plotted against the porosity (column (ii)). The points have been numbered to correspond with the numbers of the ores in column (i) ; larger circles denote the positions of Wabana and Kiruna as the exact extent of penetration is not known, and suitable corrections have been made to the values for Keloucha and Djerissa to allow for the smaller size of lump used. In general, the extent of penetration is directly proportional to

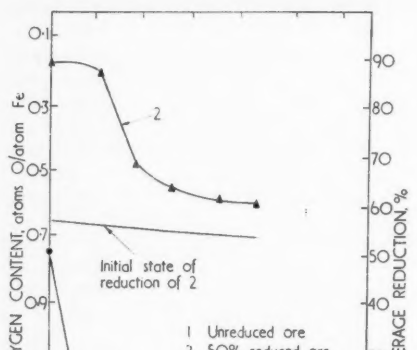


**Fig. 22—Penetration into unreduced and partly reduced Ouenza ore**

the porosity, but there are certain marked divergencies, in particular Konakry (5), Cleveland (18), Grangesberg (11), and Gellivare (12). The partial break-up of Cleveland may account for the comparatively deeper penetration, whilst the fissured nature of the Konakry allows considerably greater penetration into the lump than the porosity would suggest. The extra penetration into Grangesberg may be explained by its structure ; the quartz groundmass will suffer a volume change during heating, and the biotite (mica) crystals will split and further open up the lump to gaseous penetration. With Gellivare, the apatite and the complex silicate will undergo volume changes on heating, thus facilitating penetration of the lump ; here, however, the effect on reduction is

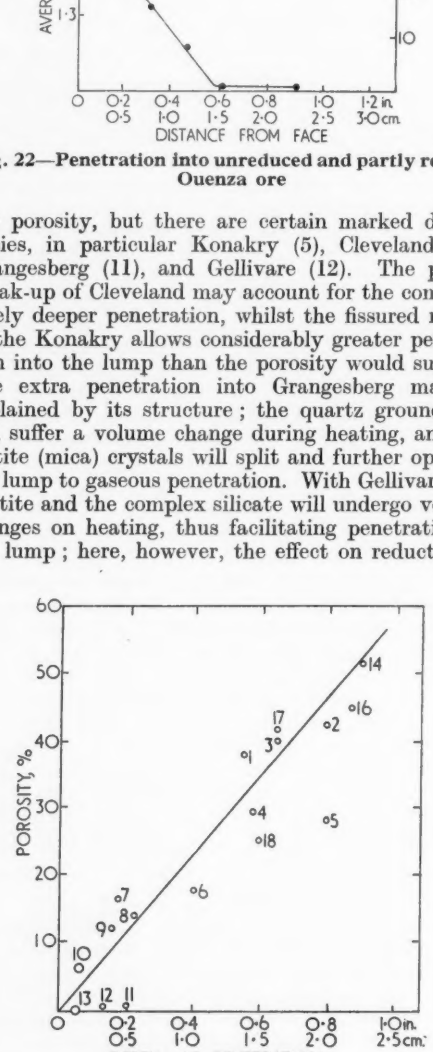


**Fig. 23—Variation of penetration with porosity**

probably less as the individual crystals are large in size (about 3–4 mm.) and regular in shape.

Assuming that chemical reaction is very fast, and considering only diffusion of CO into the matrix, reduction would be expected to take place in a series of distinct stages, each stage being governed by the $CO/CO_2$ ratio of the gas. This simple picture is complicated by the fact that diffusion of the products of reaction out of the lump is proceeding simultaneously, and thus the outer layers may be in contact with gas of a $CO/CO_2$ ratio such that considerable reoxidation can take place, causing, in the limiting case, uniform distribution of oxides and iron from the face to the limit of penetration. A third factor must also be considered when the distribution of oxygen between the oxides of iron is surveyed, *viz.*, the fact that FeO breaks down at around 550° C. to free iron and magnetite, and thus the rate of cooling of the lump will affect the relative amounts of Fe, FeO, and $Fe_3O_4$. Whilst the relative amount of ferrous iron in a range of samples proves that this decomposition is not complete under normal cooling, the appreciable percentages of free iron in the unreduced cores of the lumps containing considerable percentages of ferrous iron initially, *e.g.*, May-sur-Orne 2½–3% of free iron in core, Cleveland 5–6%, Northamptonshire Green 3–3½%, show that the breakdown is taking place.

Whilst the extent of penetration is, in general, proportional to the porosity of the ore, certain phenomena cannot be explained by this alone. In particular, one group of the most penetrable ores, Sierra Leone, Oxfordshire, Frodingham Silicious, and Northamptonshire Green, has a uniform oxygen gradient from the surface to the limit of penetration. The extent of reduction at the surface is not great and the slope is gradual; another group of ores, where the penetrability is, in general, rather less, Ouenza, Djerissa, Keloucha, Konakry, May-sur-Orne, and Cleveland, has a much larger superficial reduction with a very steep oxygen gradient through the surface layer, followed by a less steep gradient through the remainder of the reduced zone. Any difference in grain size might influence the rate at which equilibrium between the diffusing gases and the ore is established. What is rather surprising is the fact that the overall reduction is, in general, greater with the second group of ores than with the first, although exact evaluation of the extent of these differences is not possible since the oxygen gradient in the first ⅛ in. of the lump is not known with any exactitude. A similar effect was obtained with ores of like porosity—a limonite of porosity 60% and a hematite of porosity 37%—by Kalina and Joseph,[12] reducing with $H_2$.

With ores of low permeability the total depth of penetration is small, and it is not possible to assess the detail of the oxygen gradient within the lump. Also, the surface layer of the ore used for analysis, whilst only some 1 mm. in thickness, amounts to an appreciable proportion of the total reduced zone in the case of the least penetrable ores. Thus it is possible that the reduction in this layer is made up of two portions, a narrow surface layer with a high degree of reduction, and a broader inner zone reduced to a very much lesser extent. Microscopical examinations of sections of Oued Zem and Rif support this since in each case there is a narrow, rather variable, zone containing a high percentage of free iron surrounding an annulus of reduced oxides (*see* Figs. 14*b* and 15*b*).

With dry permeable ores the temperature lag at the limits of penetration might be as great as 40° C. (Fig. 10), but it is doubtful whether this will exert any large effect since the reaction taking place here will be the rapid, irreversible reduction of $Fe_2O_3$ to $Fe_3O_4$. Where water of hydration and carbonates are present, however, the effect will be much greater, and the temperature lag may be much greater and exert an appreciable influence on the extent of the reaction inside the lump (Fig. 8*c*).

Many of the more permeable home ores will probably suffer considerable disintegration on the way down the stack, thus penetration may well be complete under normal conditions. From the point of view of ore preparation in the stack, it might be possible to increase the driving rate of a furnace with such ores, but the limiting factor would probably be dust losses. The break-up of somewhat similar French ores under blast-furnace conditions has been studied by Fortado.[22]

Break-up increases greatly the overall degree of reduction of Wabana by increasing to a very large extent the surface available for gaseous attack. Reduction of Gellivare might also be facilitated by disintegration, though the nature of the individual crystals would prevent rapid reduction.

The rate of heating, especially the time spent in the carbon deposition zone, will have a considerable influence on the nature and extent of the break-up of ores, and fluctuation in furnace operation may, on occasions, be attributable to this.

The penetration of reduction into low-grade limy ores may be very considerable, owing to the open nature of the matrix after heating and the high reduction potential of gases penetrating through regions of low iron content; the extent of the reduction, however, may be limited by the temperature lag within the lump due to $CO_2$ evolution.

With rich ores of low permeability the amount of direct reduction which must be carried out in the hearth is large, and hence the heat requirement of this region will be a critical factor; thus increase of blast temperature should be most effective for such ores.

## CONCLUSION

The overall rate of heat transfer to the centre of the lumps does not vary very greatly with different ores and is not likely to be a critical factor except, possibly, in certain permeable home ores where the rate of heating inside the lump is retarded by endothermic reactions. In these ores calcination would have a beneficial effect.

In general, this work has established the fact that gas penetration into lump ores under blast-furnace conditions, and hence stack reduction, is limited by the porosity of the ore, and this factor should therefore determine the optimum size. Certain deviations from this principle have been noted and reasons for them

have been discussed. The actual sizes suggested for blast-furnace practice are comparative only, being based on a lump size of 3 in. for Ouenza. The extent to which these optimum sizes could be achieved in practice is linked with crushing efficiency and the ability to handle the fines produced.

With certain ores, wide variations in chemical and physical properties occur and, although an effort was made to secure a representative sample for investigation, the results for such ores should be interpreted in conjunction with analysis and porosity figures for the actual consignment to be used. The samples of magnetite ores employed were chosen to cover a range of ore types and thus to assess the effect of various gangue constituents.

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# Studies on Marine Fouling Organisms

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### SYNOPSIS

This paper summarizes the work which has been done on the biological aspects of the fouling problem, mainly at Millport (Firth of Clyde) during the period 1944–48. Much of this work has been published in the appropriate biological journals.

A continuation of non-toxic panel exposures, both at Millport and elsewhere, has added fresh details to what was known about the seasonal sequence of settlement and emphasized the extent of variations in density of settlement from year to year. Laboratory determinations of the sensitivity of most of the major fouling organisms to copper and mercury have revealed many complexities in this phenomenon which, it is suggested, can only be resolved by fundamental studies of the action and interaction of these poisons. Many observations have been made on the general biology of a number of animal fouling organisms. The ecology of their interrelationships, and of their relationships with marine plants, deserves further study.

After the publication of the "Report on Anti-Fouling Research, 1942–44,"[1] it was decided that further accounts of work on the fouling problem should appear in the form of separate papers contributed to the appropriate journal by the worker most closely associated with each aspect of the work. Consequently, about 30 papers have since been published, prepared, or planned. The majority of published papers dealing with the chemical aspects of work on the fouling problem have appeared in this *Journal*,[2, 3, 4, 5, 6, 7] together with some which covered general topics,[8, 9, 10] but most of the results of the biological work have been, or will be, published in the appropriate biological journals. The purpose of this paper is to summarize briefly the results presented in these biological papers.

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Biological work on the fouling problem since 1944 has developed very much along the lines which were sketched in the comprehensive report[1] and consequently a similar sub-division into topics will serve for the present account. The author is indebted to his former colleagues for their permission to quote from their unpublished work in order to make this survey as complete as possible.

## SEASONAL SETTLEMENT OF FOULING ORGANISMS

Routine exposures of non-toxic panels were maintained from the Millport rafts during the whole period that work on the fouling problem was in progress, and extensive series of exposures were also made at Birkenhead, Caernarvon, Plymouth, and Chichester Harbour. Less extensive series of exposures were made at Shoreham Harbour, Billingham, the River Wear, and South Shields. The panels exposed at Millport were examined in the greatest detail, since these could be assessed when the settlements were living ; for the other exposure sites, the panels were examined after preservation in formalin vapour, so that much of the finer detail was missing. The panels exposed at Chichester Harbour were an exception to this rule, since these were assessed, immediately after withdrawal, by Dr. Stubbings, whose help is gratefully acknowledged. These panels were again assessed (in the preserved state) at Millport.

The extension of the Millport exposures has added further details to the account of seasonal settlement given in the 1942–44 Report, but it has not modified the main sequence. Differences include the recognition of settlements of the larger brown algae (*Saccorhiza*, *Desmarestia*, and *Laminaria*) from October to February. These algae settle over this period but do not grow until March or April, when they grow very rapidly. Further observations have shown that the settlement of mussels (*Mytilus*) normally occurs rather earlier (July, August) than had been realized and that the predominance of plant fouling may not always extend so far into the summer ; the 1942–44 Report gave July as the month in which plant fouling reached its maximum, but in many years settlement in this month is predominantly animal, often consisting largely of the hydroids *Obelia* and *Tubularia*.

The prolongation of the non-toxic panel exposures has also provided further instances of variations in density of settlement from year to year. The spring settlement of *Balanus balanoides* has perhaps provided the most striking examples of such variations. Settlement during April 1943 was very light ; it was heavy throughout the corresponding month of 1944, moderately heavy, but limited to the first two weeks, in April 1945, and light in 1946, 1947, and 1948.[11] The settlement of *Tubularia larynx* also sometimes presents variations, *e.g.*, in 1945 settlement began unusually early but the population died down early in August and only reappeared in any number in mid-September, and there were signs of a similar condition two years later. Among plant fouling organisms, the heavy summer settlements of the diatom *Fragilaria* in 1942 were not repeated in the succeeding six years. Such variations may all be important in the accurate assessment of anti-fouling compositions under test, and their occurrence makes it imperative to maintain a series of non-toxic plate exposures while anti-fouling compositions are being tested.

A fuller account of the results of the non-toxic panel exposures at Millport will be presented in a separate paper, which will also include details of the other investigations carried out there in connection with these exposures, *e.g.*, effect of type of surface, area of surface, and depth of exposure on the type and amount of settlement.

Comparison of the results of non-toxic plate exposures at Millport with those made elsewhere suggests that, though fouling settlement at Millport is both extensive and varied, the density of settlement is generally rather less than that at the exposure sites on the south coast. The examination of plates exposed from the Plymouth raft (which is situated some distance off-shore, just inside the breakwater) shows an extension of the period of heavy algal settlement further into the summer, and the presence of *Plumularia setacea* as the common summer hydroid ; at Millport this settlement is replaced by one of *Obelia longissima*. The heavy settlements of the compound ascidian *Diplosoma*[1] have continued to be a conspicuous feature of the autumn fouling at this site.

The heaviest settlements have been recorded from plates exposed from the Admiralty raft in Chichester Harbour, which proved to be the most severe of the fouling sites investigated. Heavy settlements of barnacles and ascidians are characteristic ; hydroids (particularly *Plumularia setacea*), and a wide variety of green and brown algae are also common. The barnacle settlements consist of *Balanus improvisus* and the Antipodean species, *Elminius modestus*. The first published record of the occurrence of the latter on our coasts is based on examinations of Chichester Harbour plates,[12] but more recent records[13] indicate that this species is plentiful over much of the south coast. The occurrence of *Elminius modestus* is of considerable interest biologically, and Knight-Jones[14] has pointed out that, because of its tolerance of brackish water and its long settlement period (which extends over most of the summer), this barnacle may become a serious menace to the oyster fisheries of the south-eastern estuaries.

The records from Sunderland, South Shields and, to a slightly less extent, Birkenhead, emphasize the importance of the colonial Protozoan *Zoothamnium* as a constituent of estuarine fouling. The branching habit of this minute form favours the accumulation of silt, which is a conspicuous element on panels exposed at these sites. The panels exposed at Birkenhead showed the greatest density of fouling for these river stations. Corlett[15] has recently published a more detailed account of colonization at various sites in the Mersey, which demonstrates the rather surprising variety and density of settlement near Liverpool and Birkenhead.

## SENSITIVITY TO POISONS

Harris[1] has already published an account of the toxicity of a wide range of organic compounds and some inorganic compounds to red, brown, and green seaweeds and to the copepod crustacean, *Nitrocra spinipes*. These tests, carried out by Dr. Barnes and

Miss Stanbury, were made primarily to discover the toxicity of a number of organic compounds relative to that of copper sulphate or mercuric chloride. In later work on this subject, the poisons used were almost invariably the copper and mercury salts just mentioned, but their effects on a range of different plants and animals (known to be of importance as fouling forms), or on stages in the life history of one species of plant or animal, were estimated. The results of this work can be most conveniently summarized under the different types of organism used.

**Algae (Seaweeds)**

The most comprehensive work on the sensitivity of seaweeds was carried out by Miss Stanbury. The results will shortly be published in detail and it is therefore only necessary to mention them briefly.

Extended tests were made on 18 species of marine algae, all known to have occurred as fouling forms. Ten of these species were diatoms, four were green algae, and two each brown and red algae. Judged on the basis of the concentration necessary just to prevent growth, each species proved to be more sensitive to Cu than to Hg and in general the Hg/Cu ratio (wt./wt.) was 2 : 1. The range of sensitivity was considerable. Expressed in terms of Cu concentration (p.p.m.) it varied from 1·0–1·3 (the diatom *Amphiprora paludosa*) and 1·0 (the diatoms *Amphora exigua*, *Schizonema Grevillei* ; the brown alga *Ectocarpus* and the green algae *Enteromorpha* and *Ulothrix flacca*) to values of 0·38 (the diatom *Licmophora Lyngebei* ; the green alga *Cladophora* and the red algae *Ceramium rubrum* and *Antithammion floccosum*) and 0·26 (the diatom *Synedra tabulata*).

The extreme instance of sensitivity to Hg and comparative insensitivity to Cu is shown by the algal settlements which have been loosely termed ' brown mats.' Their precise identity is uncertain, some (possibly most) of these settlements are either growth stages or growth phases of the brown weed *Ectocarpus*, but other brown algae (*e.g.*, *Hecatonema*, *Myrionema*) have from time to time been observed to form these growths. They are so insensitive to Cu, that they can settle on the surface of a copper paint which has a leaching rate high enough to prevent the settlement of all other major fouling organisms, but so sensitive to Hg that they have never been observed to colonize the surface of a composition containing this poison, even if Cu is also present and the ratio Cu/Hg is 60 : 1.

Experiments using mixtures of Cu and Hg showed that ' sub-effective ' doses of Cu and Hg can, when mixed together, become poisonous. The effect of the mixtures is less than additive.

Seasonal variations in sensitivity were also detected during the course of these experiments, the algae being more resistant during the summer than during the winter months ; an effect doubtless due to the increased vigour of the plants during the summer, a phenomenon which itself results from the interaction of a number of factors.

Experiments on the effect of light intensity on the growth of seaweeds showed clearly that growth is retarded by light of reduced intensity and that, as would be expected, growth is proportional to the energy transmitted to the plant. As the intensity is rapidly reduced as light penetrates beneath the surface of sea water, growth becomes retarded at greater depths. This, as Harris[1] pointed out (p. 316P) may explain why the action of copper on fouling seaweeds varies with light intensity.

**Hydroids (*Obelia* and *Tubularia*)**

Laboratory tests on the sensitivity of the settling stages of *Obelia* and *Tubularia* to Cu indicated that both were very sensitive to this poison (median lethal concentration less than 0·1 p.p.m. for the settling stages themselves, about 0·1 p.p.m. for the recently settled individuals) but that the adult colony of *Obelia* (median lethal concentration 0·05 p.p.m.) was much more sensitive than that of *Tubularia* (median lethal concentration 2·5 p.p.m.). Though the latter result agrees with the sensitivity of these two hydroids as deduced from colonization data, the difference is unduly emphasized under laboratory conditions because colonies of *Obelia* do not remain healthy when kept in running sea water in the laboratory (even in the absence of poisons) whereas colonies of *Tubularia* remain in a healthy state under these conditions for many days. Like some other animal fouling forms, but unlike the seaweeds mentioned in the section above, both *Obelia* and *Tubularia* are more sensitive to Hg than to Cu.

Brief reference to these experiments is made in the paper by Pyefinch and Downing.[16]

**Tube-Worms (*Pomatoceros* and *Hydroides*)**

The work carried out on the biology and sensitivity of tube-worms was initiated by Dr. Mare and Miss Mott, and continued by Mr. Bishop who will later publish an account of this work.

Though the genus *Hydroides* is far more common as a ship-fouling form than *Pomatoceros*, most of the work on fouling tube-worms was carried out on the latter genus, because it occurred much more commonly on panels and other raft-exposed surfaces.

*Pomatoceros* is moderately sensitive to Cu (median lethal concentration 0·5 p.p.m. or less) but less sensitive to Hg (the median lethal concentration may be as high as 1·5–2·0 p.p.m.), thus this tube-worm agrees with the seaweeds in relative sensitivity to these two poisons. These observations confirm those made by Miss Stanbury. Sensitivity to Hg seems to vary inversely with length (the value quoted above was obtained for adult specimens).

A few observations were also made on the sensitivity of *Hydroides* to Cu and to Hg ; these suggested that this tube-worm was less sensitive to either poison than *Pomatoceros*, the median lethal concentration being greater than 3·0 p.p.m.

**Barnacles (*Balanus*)**

Work on the sensitivity of barnacles and their larvae to Cu and to Hg[17] has shown that considerable differences in sensitivity occur at different stages of the life history. To summarize these results briefly, all the nauplius larva stages are sensitive to the action of these poisons, but the cypris stage (which follows) is appreciably less sensitive to either. If cypris larvae are kept in the laboratory, their sensitivity increases

over a period of four or five days. As settlement normally takes place towards the end of such a period, this can be interpreted as an increasing sensitivity as the time for settlement approaches. It is certainly true that very low concentrations of Cu and Hg (*e.g.*, exposure to 0·1 p.p.m. Cu or 0·01 p.p.m. Hg for 12 hours) are able to prevent settlement under laboratory conditions, though they may have little or no effect on the activity of the cypris larvae. Once settlement has taken place and the metamorphosis into the adult begins, the developing barnacle is less sensitive to heavy metal poisons than at any other stage in its life history, but as soon as metamorphosis is completed, the sensitivity again increases.

Like *Obelia* and *Tubularia*, barnacles are more sensitive to Hg than to Cu, the Hg/Cu ratio (wt./wt.) being roughly 1 : 2 for the cypris larvae. Sensitivity estimations were made on *Balanus crenatus* (one of the common fouling species of northern temperate waters) and on *B. balanoides* (the common shore barnacle of this country) which does not often occur as an important ship-fouling form but did prove to be the most important component of barnacle fouling on panels exposed from the Millport rafts. These two species showed certain differences in the sensitivity of the various stages in their life history (*e.g.*, the cyprid of *B. crenatus* is much less sensitive to Cu and rather more sensitive to Hg than the corresponding stage of *B. balanoides* ; Cu and Hg appear to be equi-toxic to the adult *B. balanoides*, whereas the adult *B. crenatus* is slightly more sensitive to Cu and distinctly less sensitive to Hg). The discovery of such differences emphasizes the danger of assuming that the sensitivity of one species is closely similar to that of another species of the same genus.

### Other Invertebrates

A number of observations were also made on the sensitivity of some of the other invertebrates which occasionally colonized panels exposed on the rafts, though none were important fouling forms.

Most work was done on mussels (*Mytilus edulis*). These proved to be less sensitive to Hg than to Cu, as attachment occurred when mussels were exposed to concentrations up to 2·5 p.p.m. Hg, but attachment did not occur if the Cu concentration exceeded 1·0 p.p.m. These results (Bishop, unpubl.) confirmed those obtained by Stanbury (unpubl.) but mussels are not very convenient subjects for experimental studies of this kind since, when placed in an unfavourable environment, the shell closes. In this condition, the mussel can survive exposures to considerable concentrations of poisons (*e.g.*, 200 p.p.m. Hg) for long periods. Further, the initial sensitivity and ultimate toxic effect may be different, since mussels appear to be initially less sensitive to Hg than to Cu, but the death rate after a period of exposure to the poison is higher in Hg than in Cu solutions, probably because there are greater opportunities for Hg to become associated with the tissues.

Experiments on the Polyzoan *Bowerbankia* and the colonial sea-squirt, *Didemnum*, showed that the former is more sensitive to Hg than to Cu, though *Bowerbankia* can withstand appreciable concentrations of both elements ; the median lethal concentration for Cu is 1·2 p.p.m., whereas that for mercury is 0·7 p.p.m. (24 hours' exposure). The sensitivity of *Didemnum* to Hg appears to be about half that of *Bowerbankia*.

### General Comparisons

A review of these results clearly indicates the uniformity shown by the algae (which are all more sensitive to Cu than to Hg) compared with the disparity shown by the animal fouling organisms tested (*e.g.*, *Obelia*, *Tubularia*, and barnacles are more sensitive to Hg than to Cu, whereas *Pomatoceros* and possibly mussels are more sensitive to Cu than to Hg). Differences of this sort were perhaps to be expected, since all the plant fouling organisms belong to one group of the plant kingdom, whereas the animals belong to a variety of groups, differing widely in complexity of organization.

If the comparison is taken further, other differences become apparent. Pyefinch[9] has described a fouling sequence which expresses the order of increasing sensitivity of a number of important plant and animal fouling organisms. This sequence was deduced from observations on raft-exposed panels and it has since been generally confirmed by observations on the critical leaching levels for key members of the series.[5] If this sequence is compared with the sensitivities of plant and animal fouling organisms, determined in the laboratory, the order of sensitivity of the plant organisms agrees closely with the fouling sequence, but this is by no means true for the animal organisms.

| Plant Fouling Organisms | Animal Fouling Organisms |
|---|---|
| *Amphiprora* (1·0–1·3) | |
| *Ulothrix* (1·0) | |
| *Achnanthes* | |
| | *Tubularia* (2·5) |
| *Ectocarpus* (1·0) | |
| | *Balanus* (0·65) |
| | *Obelia* (0·05) |
| *Ceramium* (0·38) | |
| *Cladophora* (0·38) | |
| *Laminaria* | *Pomatoceros* (0·5) |
| *Desmarestia*, etc. | *Mytilus* (1·0) |

The figures in brackets roughly represent the sensitivity to copper (mg./l. Cu). The numerical data are illustrative only and are not quantitatively comparable.

The copper sensitivities of the animal fouling organisms cited are those of the adult organism, but, if the values for the settling stages or recently settled individuals are inserted instead, this does not resolve the anomaly. The physical conditions at the surface of an anti-fouling composition releasing poisons into the water suggest that the size of the settling stage might be a contributory factor. Immediately over the surface of the paint, flow will be laminar, and it is only within this laminar layer that an appreciable concentration of poison can exist, since outside this layer the turbulence of the flow will rapidly reduce the poison concentration to a negligible value.[18] If the settling stage is small enough to lie wholly within this laminar layer when first attached to the paint surface, the flow characteristics will not be disturbed and the settling stage will be exposed to the full effects of whatever poison concentration exists. If, however, it is large enough to project through this

layer when first attached, laminar flow will be disturbed by the projection, and the settling stage will be exposed to a low, if not negligible, concentration of poison. Organisms which have settling stages large enough to project through the laminar layer should therefore be able to settle on surfaces which are maintaining an appreciable leaching rate, whereas those with smaller settling stages should not. It is difficult to estimate the thickness of the laminar layer, but *Tubularia* and barnacles (which can settle on surfaces with Cu-leaching rates of 9 and 7 μg/sq. cm./day respectively) have settling stages which will project through the laminar layer, whereas the settling stages of *Obelia* and *Pomatoceros*, which are much smaller, probably lie wholly within this layer when first attached, and these genera are known to settle only on surfaces maintaining much lower leaching rates. Laboratory determinations of sensitivity may thus be misleading, since there the organisms tested are wholly bathed in the poison, whatever their size.

Though the size of the settling stage may play some part in determining the possibility of settlement for animal fouling organisms, the same condition cannot apply to plant fouling organisms since their spores are all small enough to lie well within the laminar layer. The causes of variations in the sensitivity of these organisms are unknown.

### Fundamental Work

There remains much to be learned of the more fundamental aspects of the action of Cu and of Hg on living organisms. Indications are not wanting from several aspects of the work on the sensitivity of fouling organisms, *e.g.*, the differences between *Balanus balanoides* and *B. crenatus*, the differences in relative sensitivity to Cu and to Hg between the algae and some animals, etc. Unfortunately, work on the fouling problem at Millport was discontinued before much progress could be made with more fundamental studies, but some work was done along these lines. Barnes and Stanbury[19] have described the effect of mixtures of Cu and Hg on *Nitocra spinipes* and have discussed the chemical conditions obtaining in mixtures of this kind, and Hunter[20] has published an account of detailed studies of the effects of Cu and Hg on *Marinogammarus marinus*. This amphipod appears to be virtually insensitive to Cu over the range of concentrations studied, though much more sensitive to Hg. At very low concentrations, Cu has a pronounced effect on animals undergoing Hg poisoning, but the reverse does not seem to be true. This preliminary investigation indicates that for *M. marinus*, the toxic action of Hg was exerted by a direct poisoning of the protoplasm, whereas Cu acted indirectly, by affecting some metabolic process. Indications of complexities in the action of Cu were also observed in the work on barnacle larvae.[17]

## GENERAL BIOLOGY

Work on the biological side of the fouling problem provided many opportunities for observations on the general biology of fouling organisms. Some of these have already been briefly described in the section on the seasonal settlement of fouling organisms (p. 215) and there are doubtless many which have not yet been published. The published work is summarized in the following.

### *Tubularia*

The actinula larva, the settling stage of this hydroid, probably has a very short life as a free-swimming individual. Darkness or still water seem to favour liberation and, though these larvae do not settle under laboratory conditions until about 24 hours after liberation, there is evidence which indicates that this interval is much shorter under more natural conditions in the sea. Settlement, in fact, often takes place in the immediate vicinity of the parent colony. This larva can settle on a wide variety of surfaces, including those (*e.g.*, a heavy bacterial slime or a dense covering of ' brown mats ') which are unsuitable substrata for the settlement of almost all other fouling organisms and which are frequently unfavourable for the persistence of the colony which develops. Once settlement has been completed, growth takes place rapidly, and the colony which results is itself capable of liberating larvae just over three weeks after settlement. Most of these larvae settle near their parent and it is therefore evident that colonization by *Tubularia* can become very dense.

At Millport settlement of *Tubularia* can take place from May until the end of December, but settlement is rarely heavy until the end of July. This period of heavy settlement extends through August and usually through September, but by early October most of the colonies are dying down. The polyps are attacked by a number of other marine animals, but these attacks do not seem to be the fundamental cause of the autumnal decline of *Tubularia*. It has been suggested that the decline of other hydroids (*e.g.*, *Obelia*) is due to internal factors and it seems likely that these may also be important in *Tubularia*.

Fuller details of these observations have been given by Pyefinch and Downing.[16]

### Tube-Worms (*Pomatoceros* and *Hydroides*)

Settlement of *Pomatoceros* and *Hydroides* is mainly restricted to the summer months and is heaviest in June, July, and August. The larva of *Pomatoceros* can settle on most non-toxic substrata, but it does not settle readily on a non-wettable wax surface and attachment is clearly insecure on a slimy, smooth glass surface or on a greasy surface. It seems likely that speed of water flow has a marked effect on settlement as there is indirect evidence which suggests that it only occurs over areas or at times when water movement is at a minimum. Far fewer observations were made on the settlement of *Hydroides* larvae, but an exposure of an ion-exchange resin (which had a rough, granular finish) provided evidence which strongly suggested that *Pomatoceros* could not settle on a rough surface, whereas *Hydroides* appeared to settle equally readily on rough and smooth surfaces.

Studies of the rate of formation of the calcareous tube in which these worms live show that it varies with both the age of the worms and the season of the year. Under favourable conditions the tube of *Pomatoceros* increases in length at the rate of 0·3–0·4 mm./day and reaches a length of 70 mm. in the

first year, whereas the tube of *Hydroides* increases in length by 0·5–0·6 mm./day and is about 100 mm. long at the end of the first year. There appears to be no direct correlation between rate of tube secretion and temperature of the sea water, but food supply is presumably an important factor ; the vernal increase in the rate of tube formation coincides with the spring diatom increase and the rate of tube formation decreases again in October, when planktonic diatoms are becoming less abundant. Young worms appear to secrete their tubes almost continuously, but older ones carry out this process spasmodically.

**Barnacles**

A number of observations have been made, both on adult barnacles and on their larvae, since the publication of the 1942–44 report. Observations on the occurrence of the larvae have been facilitated by the development of methods of rapid identification. Previously, the diagnosis of the specific identity of barnacle larvae depended on such minute characteristics as the number or arrangement of the spines on the limbs. As an early barnacle larva may measure less than 0·5 mm. in length, identification by such characteristics required careful dissection and microscopical study and was ill-adapted for the rapid identification of the many thousands of larvae which occur in plankton hauls at certain times of the year. Details of the characteristics used for rapid identification are given by Pyefinch.[21] The study of considerable numbers of the naupliar larvae of *Balanus crenatus*, together with observations on cultures of these larvae reared in the laboratory, has shown that this barnacle, like most others, has six naupliar stages[22] and not eight, as had been suggested by earlier workers.

Observations on the seasonal occurrence of barnacle larvae has confirmed and extended the statements made in an earlier report.[1] The larvae of *Balanus balanoides* occur in February, March, April, and May, the greatest number occurring in March and April, whereas, though the maximum number of the larvae of *B. crenatus* occur during the months just mentioned, smaller numbers occur throughout the summer. They tend to occur periodically and the tendency is especially evident in the cyprid stage, so that settlement of this species occurs periodically.

Though considerable numbers of naupliar larvae of both species may occur in the plankton during February and March, the numbers of cyprids which occur in April do not necessarily correspond ; for example, if the numbers of cyprids taken in April are compared with the numbers of earlier larvae taken in the preceding month, the results are as follows :

| Year | Apr. cyprids/Mar. nauplii |
|---|---|
| 1944 | 52 % |
| 1945 | 50 % |
| 1946 | 4 % |
| 1947 | 21 % |

Thus, in 1944 and 1945 the sequence of larval stages was reasonably successful, whereas in 1946 and to some extent in 1947, it was much less so. The reason for these differences seems to be the effect of the presence of diatoms in the water. The spring increase in diatoms usually occurs in the Clyde about the middle of March when very heavy hauls of diatoms are taken for a week or ten days, and then the numbers present decrease appreciably. This was the sequence in 1944, but in 1946 large numbers of diatoms appeared rather earlier than usual and persisted until the middle of April. This persistence was apparently unfavourable for the survival of the cyprids of *B. balanoides*. The cyprids of *B. crenatus* do not appear to be affected to nearly the same extent and the results obtained in 1947 might well have been of the same order as those obtained in 1946, had the number of *B. crenatus* cyprids been unusually high.

An attempt was also made to determine whether short-period fluctuations occurred in the number of barnacle larvae, which might be correlated with such conditions as time of day or tidal state. Except that the number of the cyprids of *B. balanoides* (and probably those of *B. crenatus*) seemed to be less abundant during the hours of darkness than during the day, no firm conclusions could be drawn from this investigation. It did, however, provide some interesting information on the efficiency of nets as a means of quantitative estimation of the plankton ; a fuller account of this is given by Pyefinch.[23] Using the results obtained in this study, it is possible to estimate the losses, by death or dispersal, during the sequence from first stage nauplius to cyprid, and it appears that, for *B. balanoides*, only about one in a hundred of the nauplii which hatch survive to the cyprid stage. According to Moore[24] a single *B. balanoides* produces an average of 13,000 nauplii. Using this estimate and combining it with other data obtained in these investigations, the numerical relations between the various stages in the life history of *B. balanoides* are :

| | |
|---|---|
| Nauplii hatching | 13,000 |
| Cyprids produced | 130 |
| Cyprids surviving metamorphosis | 26 |
| Young barnacles surviving 2 months after metamorphosis | 15 |

The estimates do not allow for deaths during the cyprid stage (which may be considerable) or for deaths of barnacles caused by the crowding which would occur later than two months after metamorphosis. If allowance is made for these factors, it may well be that only one or two of the 13,000 nauplii survive to maturity as adult barnacles.

A number of observations were made on the factors affecting the settlement of the cyprid larva. A greasy or slimy surface can virtually prohibit settlement, the presence of other organisms, particularly seaweeds such as *Enteromorpha* or *Ectocarpus* or hydroids such as *Obelia*, can be an important deterrent, and a good deal of indirect evidence was obtained that speed of water flow was an important factor. Some of the results obtained, which suggested that light was an important factor in affecting settlement, could be more satisfactorily interpreted if speed of water flow was considered instead to be the operative factor.[11]

No detailed observations were made on the growth rate of *Balanus balanoides*, though it was clear that this was greater for specimens which settled on raft-exposed surfaces (which were continuously immersed) than for those on the shore (which were only immersed at intervals). Detailed observations were made on

the growth rate of *B. crenatus*. Immediately after settlement the diameter of the base of the barnacle increases at a rate of about 0·15 mm./day and, if settlement occurs in the spring or early summer, a growth rate of this order is maintained for several months, but if settlement occurs in the late summer or early autumn, it is only maintained for a short period, as growth is negligible during the winter months. The largest specimens of *B. crenatus* measured during this investigation had a basal diameter of 28–30 mm. and it seems likely that this could be attained in the course of a single summer.

*Balanus crenatus* not only grows rapidly, but also rapidly becomes mature. Individuals which had settled in April contained well-developed nauplii by July, so that the complete sequence from settlement of parent to settlement of offspring can be completed within one season.

## DISCUSSION

It has been convenient to present the accounts of the work done on seasonal settlement, sensitivity to poisons, and general biology of fouling organisms separately, but it is at once evident that all three topics are closely interconnected. This is especially clear in the case of two topics which, being of more general scope, can best be mentioned at this point.

This interconnection is well shown in the considerations which have to be taken into account when assessing the performance of a raft exposure of an anti-fouling composition. As Pyefinch[25] has pointed out, the seasonal settlement sequence is not only one of different organisms : it is one of different organisms with different critical leaching levels, and in the Clyde the seasonal sequence is such that for a period of 6–8 weeks during June and July there may be no organism settling which has a critical leaching level greater than 5 μg. Cu/sq. cm./day. Though this is not very important when only crude exposure tests are being made, it becomes more so when more refined tests, *e.g.*, estimations of the length of anti-fouling life of paint films of different thickness, are being conducted. Further, the sequence of settlement in one season is never quite the same as that in another. Examples of this have already been given (p. 215) and it will readily be realized how important some of the variations may be, *e.g.*, the failure of the barnacle settlement, or the interruption of the settlement of *Tubularia*, since these are important landmarks in the fouling season.

The three main topics discussed in this paper can thus be shown to be interwoven if raft-exposure tests are considered, and their inter-relationship is equally clear on the larger scale and under the more practical conditions of ship fouling. Accounts have already appeared[8, 10] which illustrate this point, but in interpreting the fouling sequence on ships it is equally necessary to realize the importance of more subtle interactions between the organisms which settle. Factors such as the effect of a prior settlement of one organism on the subsequent settlement of another, the preferential settlement of one species on individuals of another species, and the extent of the effects of fresh or brackish water on settled individuals are all important if the fouling sequence is to be correctly interpreted. Some notes on such factors are given by Pyefinch.[26] These analyses of fouling samples from ships have revealed many gaps in our knowledge of the general biology of marine animals and plants, particularly in tropical and sub-tropical waters. Filling these gaps does not demand elaborate investigation or extensive equipment and it is therefore to be hoped that biologists stationed near a tropical shore, perhaps for other reasons, will realize the wider importance and usefulness of such observations.

Finally, many of the investigations summarized in this paper touch merely the fringe of the problem. The need for fundamental studies of the effect of poisons on fouling organisms has already been emphasized, but there is also much to be learned about the ecology of these organisms. In particular, the ways in which metabolites of one organism can affect another organism are only just beginning to be explored. Much fruitful work lies in this field.

## Acknowledgments

Many of the investigations described in this paper were begun when Professor J. E. Harris was at Millport and they have all been conducted during his Chairmanship of the Marine Corrosion Sub-Committee. It is a pleasant duty to acknowledge the help, encouragement, and advice which has always been so generously given to all who have taken part in the investigations.

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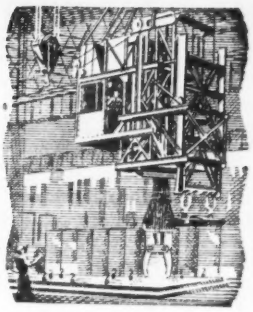
IRON AND STEELWORKS ENGINEERING

THE IRON AND STEEL ENGINEERS GROUP



# Heating and Ventilation in the Iron and Steel Industry

*By F. R. L. White, M.I.H.V.E., M.Inst.F.*

**SYNOPSIS**

Environment has a very great influence upon the general bearing and well-being of operatives. Their productive output is enhanced if they work in healthier conditions.

With a proper appreciation of the heat-producing and heat-dissipating mechanism of the human body, the problems of heating and ventilation may be regarded as constituting the maintenance of a proper thermal balance between human beings and their surroundings. It is unnecessary to provide heat for a human body; the body does that for itself. The problem is to regulate the flow of surplus heat from the human body so that it may maintain its normal temperature associated with a healthy bodily condition.

The feeling of comfort is not dependent upon temperature alone. Air movement, humidity, and freedom from contamination are all vital and inseparable factors.

In the iron and steel industry it is impossible to generalize upon the subject of heating and ventilation. Each section of the industry and each sub-division of any particular undertaking has its own individual problems, and in the majority of cases each has to be studied during production operations in order to arrive at a suitable solution. Various practical applications are suggested, and these may be separately, or in some cases jointly, adopted to provide suitable environmental conditions.

Much headway has been made in the use of mechanical aids to shift the burden from the operative, especially where excesses of temperature are to be met. Nevertheless, it is to be hoped that future research into production methods will result in greater relief from many of the present excessive conditions.

## Introduction

THE consideration of problems of heating and ventilation in industry is directed towards (*a*) the provision of healthy and comfortable environmental conditions, and (*b*) the maintenance of predetermined conditions of cleanliness, temperature, and humidity in which industrial processes may be successfully and economically carried out. These two objectives are interdependent, because conditions which are most suited for (*a*), may be unsuitable for (*b*), and *vice versa*. In industrial work, therefore, each problem must resolve itself into a quest for a reasonably healthy and economic compromise between the 'human' and the 'process' requirement factors.

Physiological research has established beyond doubt that man's general standard of health and his mental and physical alertness are vitally affected by the environment in which he lives and works. This is particularly true of environmental warmth.

## CONDITIONS OF COMFORT FOR THE HUMAN BODY

Apart from the normal requirements of air, food, and exercise, the normal healthy body must be surrounded by conditions of temperature, humidity, and air movement such that it can dispose of its surplus heat without fatigue, and a constant combination of these factors is essential for comfort.

The human body works satisfactorily only at a normal temperature of 98·4° F., and this temperature must be maintained, or serious consequences may result.

When completely at rest the body has a basic metabolism (or minimum rate of heat production) of approximately 275 B.Th.U./hr., and when walking quickly or performing heavy tasks the heat dissipation may be as high as 1400–1900 B.Th.U./hr.

The variation of skin temperature controls the rate at which heat is dissipated by radiation and convection, but there is a further outlet in the evaporation of moisture from the body, by breathing and by perspiration from the skin.

Persons can, to some extent, become acclimatized to new conditions, but there is a limit to the range

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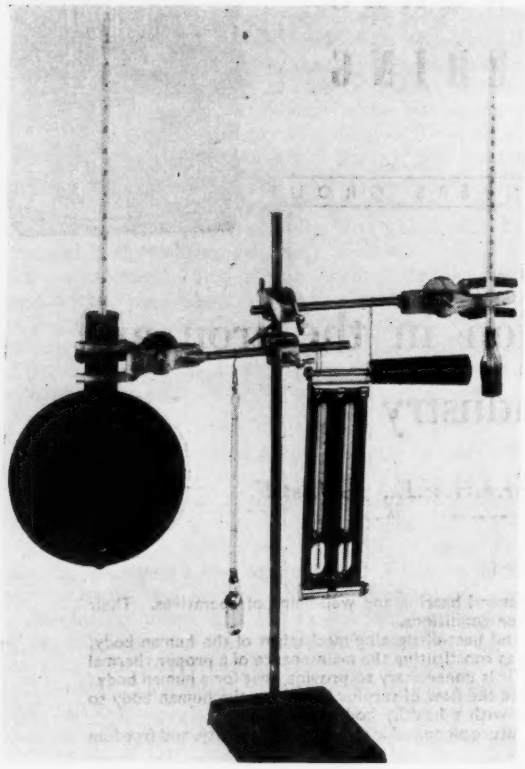

**Fig. 1—Thermometers used for the measurement of comfort conditions**

of the human thermostatic control system, beyond which it is physically impossible for human beings to endure, and to live normally healthy and active lives.

### Measurement of Comfort Conditions

Before the variable conditions of comfort can be controlled they must be measured by some known standards. Although no single instrument has yet been produced to measure comfort conditions, it is possible to obtain a measure of practical accuracy by the combined use of a globe thermometer, a kata thermometer, and a wet-bulb thermometer. These are illustrated in Fig. 1.

The globe thermometer is a hollow sphere of about 6 in. dia., coated outside with a dead mat black paint. An ordinary thermometer is inserted into the sphere so that its bulb is at the centre. The instrument is sensitive to both radiation and convection and is affected by the velocity of the air in contact with it.

The kata thermometer is an alcohol thermometer with an enlarged bulb, about $1\frac{1}{2}$ in. long $\times$ $\frac{3}{4}$ in. dia., covered with polished silver. There are two marks on the stem, one at 130° F. and one at 125° F., and a small reservoir at the top of the column receives the fluid on expansion. The fluid is heated by hot water (carried in a Thermos flask) until the column is elevated to the top reservoir. The bulb is then wiped quite dry and allowed to cool in the atmosphere of the room under test. The time taken to cool between 130° and 125° F. is noted by stopwatch for three to five tests, and the average time of cooling, divided into the kata factor, which is etched on the glass stem, gives the cooling power of the kata thermometer. With a knowledge of the air temperature, it is then possible to calculate the velocity of the air over the kata bulb.

The wet-bulb thermometer is a standard mercury-in-glass thermometer with an absorbent fabric sleeve fitted over the entire length of the bulb and extended into a reservoir containing water to maintain the bulb in a wet condition. The thermometer is whirled round at a speed of 500–600 ft./min., and when it reaches a steady state, the temperature (the sling wet-bulb temperature) is recorded. It is usual to mount a dry-bulb and a wet-bulb thermometer on one frame, fitted with a handle and pivot to enable the whole assembly to be whirled round conveniently, so that both the dry-bulb and the wet-bulb temperatures can be read simultaneously. If this is done there is no need to shield the dry-bulb thermometer as it will assume true air temperature in moving air. The depression between the wet-bulb and dry-bulb readings gives an indication of the humidity in the air.

The first practical use of an overall scale to measure environmental warmth was made in America, when Houghton and Yagloglou published a chart which they named the Effective Temperature Chart.[1] This is arranged to give an effective temperature for any combination of the three factors of dry-bulb temperature, wet-bulb temperature, and air velocity between temperature limits of 30° and 110° F. and air velocities of from zero to 700 ft./min. Later research has proved this chart to be reliable for most heating and ventilating problems, but some adjustment may be necessary for very high temperatures. Further adjustment may also be necessary to account for the effect of radiant temperature.

Vernon and Warner[2] introduced a further adjustment by using readings of the globe thermometer instead of the dry-bulb thermometer; the chart is substantially the same as the Effective Temperature Chart, and is shown in Fig. 2.

As an example of its application, for a globe thermometer temperature of 76° F. and a wet-bulb temperature of 69° F., corresponding to a relative humidity of 70%, the corrected effective temperature is 73° F. when the air movement is at 20 ft./min. Similarly, with a globe thermometer temperature of 80° F. and a wet-bulb temperature of 64° F., corresponding to a relative humidity of 41%, the same effective temperature of 73° F. is recorded for the same air movement. Thus, a rise of 29% in the relative humidity must be accompanied by a drop of 4° in dry-bulb temperature to obtain the same feeling of warmth for the same air movement.

If the air velocity is increased to 1000 ft./min. for the same wet- and dry-bulb temperatures, the effective temperature in the first case drops from 73° to 65·8° F., and in the second instance from 73° to 63·4° F. In other words, if the velocity were increased from 20 to 1000 ft./min. a person in those conditions would experience a difference in the feeling

of warmth equivalent to a drop of 7·2° F. in the first case and 9·6° F. in the second case.

### Comfort Conditions in Industry

A considerable amount of research has been carried out to establish comfort zones which will satisfy the majority of persons working in reasonably light industries, but no substantial headway has yet been made in the heavier industries.

In his investigations of thermal environment in industrial buildings, Dr. Bedford[3] suggested, as a standard, conditions in which not less than 70% of the subjects were "comfortable," and as a result of his tests, he suggested the following scale of temperature for winter conditions in Britain, set out in terms of different measures of warmth :

| Scale of Warmth | Temps. (°F.) between which not less than 70% of subjects were comfortable |
|---|---|
| Equivalent temperature | 58–66 |
| Globe thermometer temperature | 62–68 |
| Effective temperature | 57–63 |
| Air temperature (dry bulb) | 60–68 |

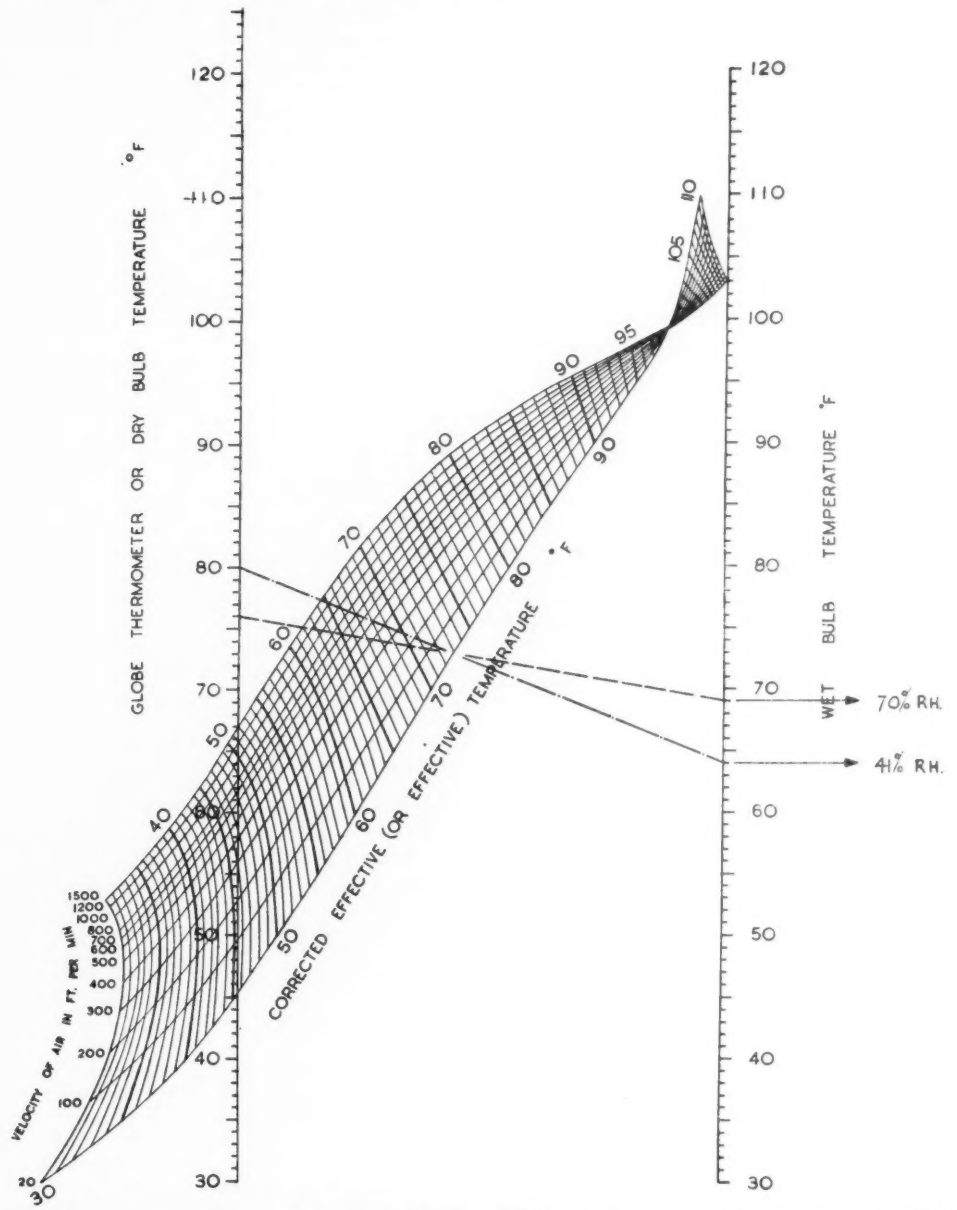
GLOBE THERMOMETER OR DRY BULB TEMPERATURE °F
WET BULB TEMPERATURE °F
CORRECTED EFFECTIVE (OR EFFECTIVE) TEMPERATURE °F
VELOCITY OF AIR IN FT. PER MIN.
70% R.H.
41% R.H.

**Fig. 2—Chart showing normal scale of corrected effective (or effective) temperature. (Based on chart published by Medical Research Council, War Memorandum No. 17, and reproduced by permission of the Controller of H.M. Stationery Office)**

The conditions refer to subjects engaged upon reasonably light work, but for those engaged upon heavy work, lower temperatures, of the order of 52–58° F. effective, or 55–60° F. air temperatures, would be more desirable.

Within the range 55–68° F. dry-bulb temperature, and air movement not greater than 50 ft./min., humidity does not have such a marked effect as with temperatures above 70° F. and velocities of over 100 ft./min. Sensations of warmth appear, then, to be more closely associated with the wet-bulb temperature.

In 1945 Eichna[4] and others carried out investigations which showed that, for average conditions, wet-bulb temperatures of about 85–87° F. were limiting conditions for tolerable comfort during exertions of short duration. The conditions were excessive, and were used only as a means to test endurance, but the results give a very informative pointer to the limits which might be encountered in industry.

Fleisher[5] and his co-workers reported in 1939 that rises in body temperatures of ½°, 1°, 1½°, and 2° F. are accompanied by increases of pulse rate of 12, 25, 38, and 70, respectively, and recommended that no person should be subject to any conditions of employment which would cause a maximum temperature rise in the body of 1½° F. during the period of exposure. This was given as the extreme limit, and an average rise of ½° F. was quoted for general adoption. On the basis of ½° F. body-temperature rise, the evidence quoted was that, for atmospheric conditions of 95°, 90°, 87·1°, 85·6°, and 84·6° F. effective temperatures, the subjects could be exposed for ½, 1, 2, 3, and 4 hr., respectively, without distress.

It is unfortunate that this valuable research work was interrupted in 1939, for it then appeared that important developments in the codifying of environmental warmth in industry would shortly be reached.

## HEATING AND VENTILATION PLANT

In 1946, the author visited a very large steelworks in Germany and noted the complete absence of heating and ventilating equipment. The German engineers explained that as this was a completely new plant, the heating and ventilating problems could not be anticipated. It was therefore decided to put the steelworks plant into production, and to design the heating and ventilating equipment on the basis of data collected from observations made under working conditions.

The iron and steel industry covers such a wide field of activity and presents problems of such diversity that it is impossible to generalize upon the type of equipment required.

Existing plants are based on the two principles of heat distribution, (*a*) convection and (*b*) radiation. But before these plants are described, mention should be made of air-conditioning plant, which is the only type which will control temperature, humidity, air movement, and filtration at one and the same time. At present it is mainly in use in cinemas, theatres, large hotels, and such places where numbers of persons congregate in comparatively congested circumstances, and where it may be necessary to remove considerable quantities of heat and body moisture. There are also many industrial processes and scientific experiments for which it is necessary to control the temperature and humidity conditions.

The plant comprises a cooler battery, a preheater battery, a spray chamber, a set of scrubber and eliminator plates, a main heater battery, a fan, and a system of ductwork to direct the air to the positions required within the building. The heater and cooler batteries are constructed very much on the same lines as a car radiator, but on a larger scale.

It is a fundamental fact that for every condition of the atmosphere, there is a related temperature at which, if the air is cooled, it will become saturated, or will reach its dew-point temperature. If the outside air is hotter than the dew-point temperature of the room, it must be cooled before saturation ; that is the duty of the cooler and refrigeration plant. If the outside air is cooler, then it must be heated before saturation, and that is the function of the preheater battery. The operations are controlled to very close limits by automatic control gear, so that if the conditions change, the necessary adjustments are automatically made in the humidifier, and/or in the heater or cooler arrangements, with remarkable sensitivity and accuracy.

Unfortunately, because of economic considerations, air conditioning has not such a wide appeal as it deserves. However, other forms of heating and ventilating, with not the same control over environmental conditions, are quite adequate for their particular purposes.

### Plenum Plant

The so-called plenum plant fulfils a useful demand for heating and ventilating in larger types of shop. It has a centrifugal fan, a heater battery warmed by steam or hot water, and a system of ducts for the distribution of the warmed air. If the atmosphere round the building is dusty or otherwise contaminated, an air filter is added to the equipment.

The fan draws air through a prepared fresh-air intake (on which would be fitted the filter) and forces the air over the heater battery, thereby raising its temperature to a predetermined figure. The warmed air is delivered through the distributing system and is discharged into the building at a specified rate, and as it cools down to the building temperature it supplies heat to offset the losses through the building fabric, at the same time ensuring adequate ventilation.

This type of plant, if properly designed, can give perfectly satisfactory results, provided that a reasonable proportion of the air can be recirculated, so conserving as much heat as possible. It tends to become uneconomical if the air is discharged to the atmosphere.

No account is taken of the humidity conditions, and hence complaints might be made of insufficient heating, even when the shop thermometer indicates a temperature of, say, 60° F. For example, suppose that the outside temperature is at about freezing point, with relative humidity of 70%—not an unusual condition—and air is drawn in and heated to, say, 60° F.; the relative humidity would be about 24%, which is an uncomfortably low figure, and would give

rise to a feeling of chilliness in some occupants, despite the temperature of the shop. Nevertheless, wherever ventilation is of primary importance, plenum schemes may well be satisfactory.

### Unit Heaters

Unit heaters have had a wide application in the past few years. They consist of a heater battery, warmed by steam, hot water, and to a lesser degree by gas or electricity, together with a fan and a set of directional louvres. The units are suspended in the shops and the louvres are directed so as to deliver the warmed air towards the occupied spaces.

Generally, the air is recirculated, but fresh air may be drawn from outside, or a mixture of fresh and recirculated air may be used.

The success of a unit heater scheme lies in having the units designed specially for the actual conditions under which they will be required to work.

Unit heaters provide the turbulence in the air which is so necessary for overcoming the feeling of stuffiness. However, the extent to which such turbulence can be endured is naturally limited, and such circumstances must be taken into account when siting the units.

### ' Radiators ' and Convection Units

The older and more conventional types of heating installations include plain and gilled pipes fixed at convenient positions, so-called radiators, built up in sectional form of cast iron or steel sections, and the cabinet type of converter units which are, in effect, gilled pipes in miniature air tunnels. These types of installation still have an important place in the field of heating, though their application may be limited.

### Radiant Heating Systems

The foregoing types of installation are all convection units, and although they have many attractive advantages, particularly economic, much of their usefulness is defeated by the difficulty of ducting the conditioned air just where it is needed. For the steel-framed type of building, which is often extremely wasteful of heat, the alternative form of radiant heating offers several advantages.

There are four types of radiant panel installations, each with its particular application :

(1) To establish comfortable conditions in local areas within a large unheated building
(2) To supplement background heating
(3) To heat an entire building
(4) To overcome cold wall or ceiling effects.

The first type may be applied where the density of occupancy is so low that it is not economical to heat an entire building. Local panels direct heat to the occupants whose activities are localized in restricted areas.

Supplementary panels can be employed where a building is used for various types of process. Thus a low background heat may suffice for the majority of the workers, and this could be supplemented by radiant panels for the operatives doing jobs of a more sedentary type.

Fundamentally, in a true radiant panel system the designer aims at obtaining the maximum direct uniform radiant energy exchange between the panel and the person. In practice, it is impossible to prevent some heat exchange by convective air currents passing over the heating surfaces, and therefore every radiant heating system constitutes a compromise between the objective of true radiation and the alternative of a conventional convection system.

### Low-Temperature Panel Systems

Low-temperature radiant panel heating has found some favour over a number of years, and takes the form of embedded coils through which hot water is circulated at temperatures of about 100–130° F. The pipes are built in as the building construction proceeds, and plastering of special specification, with scrim cloth trowelled into the finishing coat, is applied over the surfaces which contain the embedded coils, to prevent hair cracks in the plaster. The building fabric thus becomes a reservoir of comparatively low-grade heat which is transferred mainly by radiation at temperatures lower than the normal body temperature.

In buildings with low-density occupancy, this form of heating would suffice with normal window openings, but in buildings of high-density occupancy, it would have to be supplemented by mechanical ventilation. Light steel-framed buildings with steel or asbestos sheet coverings or thin brick fillings would be outside the scope of this application, and so the medium- and high-temperature radiant panels are being developed more particularly for this type of structure.

### Medium- and High-Temperature Panel Systems

The panels are fabricated by the attachment of sheet steel plates to sinuous or grid-type pipe coils, through which high-temperature hot water or steam can be circulated, and the panels thus become a source of high-temperature radiant heat.

The high-temperature rays are transmitted to the general building fabric and contents, and to the occupants, and the building eventually reaches a steady state at a temperature rather lower than that of the conventional type of installation. Despite this apparently lower temperature, the actual heating effect from a physiological viewpoint is higher and much more satisfactory.

## APPLICATIONS IN THE IRON AND STEEL INDUSTRY

Heating and ventilating equipment in the iron and steel industry embraces all the types of plant already described. Many of the processes involved in the production of iron and steel present conditions of extreme discomfort for the operatives, but with the increasing use of mechanical aids, conditions are improving considerably.

### Protection from High-Temperature Radiant Heat

The most difficult problem of all is high-temperature radiant heat, which is encountered in so many of the operations. The only way of dealing with this is by radiation shields, but these are often ruled out because they prevent proper access to the plant for operational purposes. If operatives wear protective clothing, it adds to their discomfort by preventing normal radiant and evaporative cooling from their bodies.

There seems to be little that can be done for the operative whilst he is actually engaged upon such tasks, but more could be done to reduce his physical strain when resting. Considerable relief can be given

by the provision of air-douching nozzles on flexible hoses which the men can use personally when overheated. Great care must be exercised when designing these douches to ensure that the air is at the correct temperature, otherwise severe chills and rheumatic diseases may be contracted.

Sickness records published by Vernon and Rusher[6] show that steel melters had a sickness rate of 23% greater than that of the whole of the workers, and puddlers 20% greater. Both grades of operatives suffered excessively from rheumatism and respiratory diseases, because they were far more liable to chills contracted during resting or periods of lighter work.

It is hoped that further strides will be made in the future towards the increasing use of mechanical aids, so as to shift the burden from the operative as far as is practicably possible under such excessive conditions. Much progress had been made towards this end in the iron and steel foundry sections of the industry.

**Conditions in Foundries**

Two important departmental reports dealing with the welfare of workers in the industry have recently been published, one dealing with dust in steelfoundries,[7] and the other with conditions in ironfoundries.[8] The former lays particular emphasis on dust hazards from stripping and fettling of steel castings, but the latter, known more widely as the Garrett report, has a much broader scope. Its findings are likely to have considerable repercussions within the ironfoundry industry, for it states that certain recommendations are made with a view to their adoption throughout the industry. The main points which concern the environmental condition of the operatives are :

(*a*) The elimination of all open fires and stoves which discharge products of combustion direct into the foundry

(*b*) The maintenance of a general minimum shop temperature of 50° F. when the outside temperature is 30° F., with the proviso that a temperature rise of 20° F. above the outside temperature shall be maintained when the outside temperature is below 30° F.

(*c*) The suppression and removal of all dust, vapour, and fumes at the point of origin. This refers to all operations, including pouring, knocking-out, sand handling, fettling, and core making and baking

(*d*) Insulation of buildings to prevent excessive heat losses

(*e*) The provision of washing and bathing facilities.

If proper attention is given to the collection of dust and fumes at the point of origin, so far as is practicable, the question of general ventilation of the foundry is reduced to minimum proportions, such as may be experienced in any factory or workshop.

The control of dust entails the movement of proportionate quantities of air, through which medium the dust is conveyed, and as this air is, in the first instance, mainly drawn from the foundry, it must be replaced by external air. In winter time this introduces a heating problem, and the extent of it will depend entirely upon local considerations. These considerations centre mainly around the type of production plant installed and the amount of mechanization employed, but it may generally be said that dust control should be applied to all knockouts and sand screens, all points on the conveyor where knock-out sand is transferred from one belt to another, and at all other points in the sand-handling plant where dry sand is liable to agitation in any form.

The need for dust-control equipment will vary with the type of product and the method of core-bonding, and final recommendations can be made only when all these complex considerations have been studied.

So far as the heating problem is concerned, the air which is introduced into the foundry should be at a temperature somewhat higher than the shop temperature, or the operatives will suffer from chills.

In large foundries it is advantageous to introduce fresh air through plenum-type plant, and the installations may comprise two or more self-contained units to keep the plant and ducts within reasonable dimensions, and to give greater flexibility in operation. If the air movement is small, compared with the total cubic content of the buildings, heating may well be effected by radiant panels, preferably heated by steam or high-temperature hot water.

From the heating and ventilating point of view, floor moulding foundries are more difficult to deal with than mechanized foundries, and if conditions as envisaged in the Garrett Report are to be maintained under present methods of production, the cost is likely to be too heavy to be economical.

Obviously the major considerations would be fume and vapour control, and as this can be accomplished in floor moulding foundries only through the medium of a general ventilation scheme, the amount of air involved to procure the requisite dilution could only be introduced by means of a balanced system. This comprises a plenum inlet plant introducing warm fresh air, and an extraction plant removing the vitiated air from points as near as practicable to the source of dust and vapour. This system would, of course, deal with both heating and ventilation.

No installation of this type is entirely satisfactory unless it is designed to draw away fumes, vapour, and dust from the operatives, and in many floor foundries this would appear to be an almost insuperable difficulty, unless it could be carried out with floor ducts and stand pipes with grilles near to the floor moulding alleys. In many instances this is not practicable, and the most that can be done under present methods of production is to provide a system of general ventilation to dilute the concentration of dust and fume to a practicable minimum.

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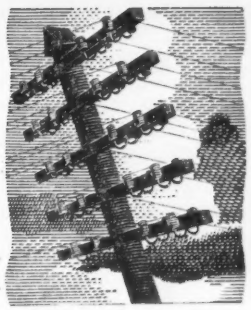
# N E W S



ANNOUNCEMENTS AND NEWS OF SCIENCE AND INDUSTRY

## THE IRON AND STEEL INSTITUTE

### Binding Cases and Holders for the Monthly Journal

After the issue of binding cases for vol. 163 (1949), binding cases will no longer be supplied free but will be available at an annual charge of 10*s*. 6*d*. per set of three, to cover the twelve monthly issues of the *Journal*. This charge will include indexes, packing, and postage.

Arrangements have been made for binding Members' *Journals* at a charge of 8*s*. 6*d*. per volume ; this charge includes packing and postage to Members' addresses, but excludes the cost of the binding case.

Stout holders have been designed into which up to four issues of the monthly *Journal* and one index may be inserted and kept for convenient handling ; single issues of the monthly *Journal* can be inserted or removed from the holders without difficulty. These holders are available on request at a charge of 7*s*. 6*d*. each, including packing and postage.

### Rolling Mill Bibliography

A bibliography on The Rolling of Iron and Steel, No. 15 in the Institute's series, has been prepared by the Library and Information Department. The bibliography covers, very extensively, the period between 1920 and 1947, but selected references to literature published before 1920 are included. It also includes references to the manufacture of seamless tubes.

Bound, duplicated stencilled copies, in two volumes totalling 457 foolscap pages, may be obtained on application to the Secretary at 4 Grosvenor Gardens, London, S.W.1, price 25*s*. (15*s*. to Members).

## NEWS OF MEMBERS

▶ Mr. G. L. Bailey, Director of the British Non-Ferrous Metals Research Association, has been elected a Member of Council of the Institute of Metals.

▶ Mr. V. T. Balasubramanian, now undergoing a training course in this country, has been awarded the degree of B.Sc. in Metallurgy.

▶ Mr. C. S. Bell, now engaged on research for the Coil Spring Federation Research Organization at the University of Sheffield, has been awarded the degree of Ph.D. of the University.

▶ Mr. D. F. Campbell, Member of Council, has been appointed Chairman of the Metallurgical Engineering Committee of the Institute of Metals.

▶ Mr. W. Castledine has joined the staff of Messrs. Salopian Engineers Ltd., Whitchurch, Salop., as Planning Engineer and Apprentice Supervisor.

▶ Mr. P. Clark has left Messrs. Thomas Bolton and Sons, Ltd., Widnes, to take up a position at Abadan, Iran, with the Anglo-Iranian Oil Company, Ltd.

▶ Mr. P. R. Dhar is now at the Standard Steel Works of the Baldwin Locomotive Works, Burnham, Pa., U.S.A.

▶ Mr. A. H. Dixon of Ashmore, Benson, Pease and Co., Africa (Pty.) Ltd., has returned to the United Kingdom from Johannesburg.

▶ Mr. L. C. Grimshaw has taken up an appointment as Metallurgist with the Firth Sterling Steel and Carbide Co., McKeesport, Pa., U.S.A.

▶ Mr. J. H. Groocock has been appointed Chief Electrical Engineer at the Abbey, Margam, and Port Talbot Works of The Steel Company of Wales, Ltd.

▶ Mr. W. G. Hull has resigned from his position of Metallurgist at Messrs. Southern Forge Ltd., to take up an appointment as a Research Investigator with the British Welding Research Association.

▶ Professor R. F. Mehl has been appointed a member-at-large of the National Research Council of the U.S. National Academy of Sciences.

▶ Professor H. O'Neill, of University College, Swansea, has been elected a Vice-President of the Institute of Metals.

▶ Dr. L. B. Pfeil, Director of Messrs. Henry Wiggin and Co., Ltd., has been elected a Member of Council of the Institute of Metals.

▶ Mr. F. W. Robinson has been appointed London Director of Thomas W. Ward Ltd., Sheffield. His office is at Brettenham House, Lancaster Place, London, W.C.2.

▶ Mr. M. N. Saxena has left the Gwalior Thermalite Corporation Ltd., Kanpur, and joined Messrs. Martin Burn Ltd., Burnpur, as a Government of India Scholar.

▶ Mr. J. Short is now at the Admiralty Materials Laboratory, Metallurgical Division, Holton Heath, nr. Poole, Dorset.

▶ Mr. H. B. Sinclair has left the Nelson Research Laboratories of the English Electric Co., Ltd. to take up an appointment as Works Metallurgist at the Stafford Works of the same Company.

▶ Mr. W. C. Smith has been appointed Works Manager at the East Moors Works of Guest Keen Baldwins, Ltd., Cardiff, in succession to Mr. J. B. Deakin.

▶ Professor F. C. Thompson, of Manchester University, has been elected a Vice-President of the Institute of Metals.

▶ Mr. N. R. Tomlinson has left the Corby works of Messrs. Stewarts and Lloyds, Ltd., to join the Export Packing Service Ltd., of Sittingbourne, Kent.

▶ Mr. T. Watson, Sales Director of General Refractories Ltd., Sheffield, has been elected President of the Silica and Moulding Sands Association.

▶ Mr. D. H. Wilkinson has taken up an appointment as Metallurgist with the Bristol Aeroplane Co., Ltd.

### Obituary

Mr. Rowland Village, of the Alloy Steel and Iron Company, Sheffield, on 25th March, 1950.

Mr. H. Hopkins, of Messrs. Guest Keen Baldwins Iron and Steel Co., Ltd., on 12th March, 1950.

Mr. J. E. Rippon, of the English Steel Corporation, on 14th April, 1950.

Mr. A. WRIGHT, former General Works Manager of the Bilston Branch of Messrs. Stewarts and Lloyds, Ltd., on 11th January, 1950.

## CONTRIBUTORS TO THE JOURNAL

**H. A. Sloman, M.A., F.R.I.C., F.I.M.**—Senior Principal Scientific Officer in the Metallurgy Division of the National Physical Laboratory.

After serving in the Royal Air Force, during the First World War, Mr. Sloman read Natural Sciences at Emmanuel College, Cambridge University. He graduated with first-class honours, taking Parts I and II of the Natural Sciences Tripos.

In 1924 he joined the scientific staff of the Metallurgy Division of the National Physical Laboratory, of which he is still a member. During the next few years he undertook research on the physical properties of glass, and was connected with the early development of refractories made from pure metallic oxides. He then began work on the production and properties of pure beryllium and its alloys, and became interested in special melting and high-vacuum techniques applied to metals. Later, Mr. Sloman turned his attention to the subject of gases in metals, a subject with which he is still intimately concerned. He is probably most widely known for his exhaustive study and development of the vacuum-fusion method for the determination of oxygen, hydrogen, and nitrogen in steel, for its extension to a large number of non-ferrous metals, and for the introduction of the now very widely used vacuum-heating method for hydrogen. He has played a very active part in the work of the Gases and Non-Metallics Sub-Committee (formerly the Oxygen Sub-Committee) of the British Iron and Steel Research Association, since its formation in 1935.

In 1948 Mr. Sloman was made a Senior Principal Scientific Officer (Personal)—a grade recently created in the scientific Civil Service, and awarded for outstanding research records—and is now in charge of teams working on problems connected with the physical chemistry of steelmaking, on the production and properties of new metals, such as titanium and vanadium, and on metallurgical aspects of atomic energy.

*Mr. Sloman was co-author of a paper published in the May issue of the Journal.*

**G. T. Harris, M.A., F.Inst.P.**—Research Manager of Messrs. William Jessop and Sons, Ltd., and J. J. Saville and Sons, Sheffield. Mr. Harris was educated at Lincoln School and at Christ's College, Cambridge University, where he obtained an honours degree in Natural Sciences (Pt. II, Physics). He joined the Research Department of Messrs. William Jessop and Sons, Ltd., in 1938, and was responsible for the organization of a section to test and develop high-temperature materials. He was promoted to Research Manager in 1947.

R. Guenot

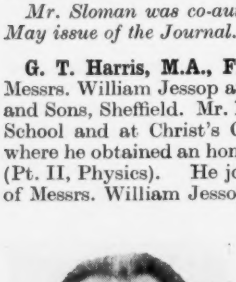
F. R. L. White

Mr. Harris is active in his support of the work of the British Iron and Steel Research Association. He is Chairman of the Non-Destructive Testing Sub-Committee and a member of the Melting and Metallurgical Committee, both of the Steel Castings Division, and a member of the Metal Physics Committee of the Metallurgy Division. He also serves on other Committees of the British Electrical and Allied Industries Research Association and the British Standards Institution.

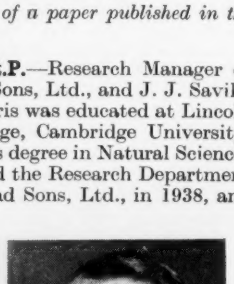
**H. C. Child, B.Sc.**—Senior Research Metallurgist at Messrs. William Jessop and Sons, Ltd., Sheffield. Mr. Child was born in Glasgow in 1924. He received his early education at Hillhead High School and then at Glasgow University and at the Royal Technical College, Glasgow. He obtained the degree of B.Sc. in 1946, taking honours in metallurgy and chemistry. He took up his present appointment with Messrs. William Jessop and Sons, Ltd., in 1945, and has since specialized in the development of heat-resisting materials and research on allied problems.

**T. A. Canning**—A member of the staff of the Chemistry Section of the British Iron and Steel Research Association's laboratories at Sketty Hall, Swansea. Mr. Canning was born in 1920 and was educated at Pontardawe Secondary School and at Woolwich Polytechnic. Before taking up his present position he was employed at the Pontardawe Steelworks of Messrs. Richard Thomas and Baldwins, Ltd., and at the Armament Research Department, Woolwich.

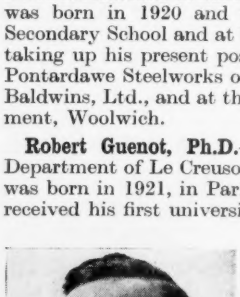
**Robert Guenot, Ph.D.**—A member of the Research Department of Le Creusot Works, France. Dr. Guenot was born in 1921, in Paris, where he was educated and received his first university training in pure science.



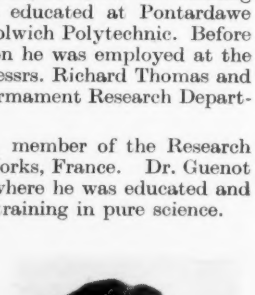
H. A. Sloman



G. T. Harris



H. C. Child



T. A. Canning

After the Second World War he entered the Ecole Centrale where he received metallurgical training under Professor A. Portevin. At the end of 1945 Dr. Guenot came to England as a British Council Scholar, and he entered Sheffield University as a post-graduate research worker. He received the degree of Ph.D. in 1949, and then returned to France to take up his present appointment.

**P. L. Chang, B.Sc., Ph.D.**—A research worker in the Metallurgy Department of Sheffield University. Dr. Chang was educated at Pei-Yang University, Tientsin, and at Sheffield University, where he obtained the degree of Ph.D., and was awarded the Brunton Medal, in 1949, for metallurgical research. He is at present engaged on research on hydrogen diffusion in iron alloys.

**B. Fang, B.Eng., Ph.D.**—A student at the works of the English Steel Corporation, Sheffield. Dr. Fang graduated at the University of Yunnan in 1941. He then came to England, where he carried out a post-graduate study at Sheffield University from 1943 to 1947. He joined the English Steel Corporation in 1948 to study process metallurgy.

**F. R. L. White, M.I.H.V.E., M.Inst.F., M.Amer.Soc. H.V.E.**—Chief Technical Officer in the Heating and Air Treatment Division of the Brightside Foundry and Engineering Co., Ltd. Mr. White was educated at Birmingham Central Secondary School, where he won a scholarship and took first prize in Mechanical Engineering and Physics. In 1914 he joined the Electrical Engineering Department of the Birmingham Municipal Technical College. After returning from service in the Royal Naval Air Service, from 1915 to 1919, he joined his father's business as a heating and ventilating engineer. He left in 1929 to join the staff of Henry Lea and Son, Consulting Engineers, and was responsible for a large part of the design of the engineering services for the Birmingham Hospitals Centre.

In 1932 Mr. White was appointed Technical Assistant (Heating and Ventilating) in the Birmingham Branch of the Brightside Foundry and Engineering Co., Ltd. He was promoted to Assistant Branch Manager in 1944 and received his present appointment in 1947.

Mr. White has been active in the professional side of his business. He is a member of Council of the Institution of Heating and Ventilating Engineers and a member of the Board of Governors of the National College of Heating, Ventilating, Refrigeration, and Fan Engineering. In 1946 he was nominated by the Association of Heating, Ventilating, and Domestic Engineering Employers to join a group of investigators visiting Germany to study German practice in heating and ventilating engineering. Last year he was invited to serve on a Joint Committee of the Institute of Heating and Ventilating Engineers, the D.S.I.R., the Medical Research Council, and the Factory Department, to study atmospheric conditions in factories.

## IRON AND STEEL ENGINEERS GROUP

### Engineering Committee

Mr. C. H. T. Williams, of the Park Gate Iron and Steel Co., Ltd., has agreed to become Chairman of the Engineering Committee, in place of Mr. W. F. Cartwright, who has been Chairman since the formation of the Committee in 1946.

### Engineers Group Meeting

The Thirteenth Meeting of the Group will be held at 4 Grosvenor Gardens, London, S.W.1, on Wednesday, 28th June, 1950. The programme is as follows:

10.30 A.M. to 12.45 P.M.—Discussion on the papers "*Lighting in Iron and Steel Works*," by G. W. Levey, and "*Heating and Ventilation in the Iron and Steel Industry*," by F. R. L. White.

12.45 P.M. to 2.0 P.M.—Buffet Luncheon.

2.0 P.M. to 4.30 P.M.—Discussion on a paper on "*Water Supply and Distribution in Iron and Steel Works*," by J. L. Daniel.

The first two papers are published, respectively, in the May and June issues of the *Journal*. Copies of the third paper are expected to be available before the meeting.

## BRITISH IRON AND STEEL RESEARCH ASSOCIATION

### International Co-operation in Corrosion Research

At the invitation of the British Iron and Steel Research Association, six Belgian scientists, representing Commission No. 4 of the Belgian Association for the Study, Testing, and Use of Materials (ABEM), toured Britain from 11th–14th April to inspect installations for corrosion research.

The tour included a visit to the Corrosion Section of the Chemical Research Laboratory, Teddington, and to BISRA's exposure stations at Brixham and Derby. ABEM, which is concerned with protection against corrosion, roughly corresponds to BISRA's Protective Coatings Sub-Committee, and the Belgian representatives were able to meet some of their British counterparts and to discuss means of collaboration.

### Pilot Steelworks Plant

The Association is to build a new research station on a 2½-acre site at Hoyle Street, Sheffield, to house a pilot steelworks plant to try out research results before application to the full-scale production plant.

Steel melting will be carried out in a 10-cwt. capacity electric-arc furnace which will be used for work on sulphur elimination and on problems of electric furnace practice.

The installation of a high-speed 14-in. 4-high cold-strip rolling mill will enable further investigations to be made into such things as 'roll force.'

There will also be an experimental wire-drawing plant in the new buildings, where new processes can be tested before going to the production plant.

The choice of Sheffield for this research establishment follows the Association's policy of placing its modern scientific facilities in the main industrial centres. This close contact between the industry and scientists ensures that problems can be communicated as soon as they arise, and that research programmes can be readily modified to meet the changing needs of industry.

## THE INSTITUTE OF METALS

At a luncheon at the Café Royal, Regent Street, London, on Wednesday, 29th March, held in connection with the Annual General Meeting, the President of the Institute of Metals, Mr. H. S. Tasker, presented the 1950 Institute of Metals Medal in platinum to the distinguished French metallurgist Professor Albert Portevin, in recognition of his eminent services to the science and practice of non-ferrous metallurgy. The French Ambassador was present at the ceremony.

## INSTITUTION OF METALLURGISTS

### Metallurgical Exhibition

Under the title of "Metals in the Service of Mankind" the Institution of Metallurgists has organized an Exhibition which will be opened by H.R.H. Princess Margaret on the 6th July, 1950. The Exhibition, which is to be held at the Science Museum, Exhibition Road, South

Kensington, London, S.W.7, will be open to the public from the 7th July to the end of September.

The Exhibition will consist of 21 stands on which all the important industrial metals, the rarer metals, and the precious metals, are represented. Some attention will also be given to such subjects as welding, metal finishing, metal testing, and electrodeposition.

In connection with the Exhibition a handbook is being prepared which will contain a short guide to the exhibits and a full section on the "Story of Metals" suitable for the general reader. The handbook will be available at the Exhibition or from the Institution of Metallurgists, 4 Grosvenor Gardens, London, S.W.1, price 1*s*.

## NEWS OF SCIENCE AND INDUSTRY

### Technical State Scholarships

The Ministry of Education has announced that the number of Technical State Scholarships available in 1950 is to be increased from 100 to 120.

Eighty of the scholarships will be available to candidates under the age of 20 on 31st July next, and up to 40 scholarships will be available for candidates aged 20 or over at that date. There is no upper age limit for the latter awards.

The scholarships will be offered to students to enable them to pursue full-time degree courses—normally leading to an honours degree—or courses of equivalent standard at Universities, University Colleges, or Establishments for Further Education.

Candidates must have completed a course leading to an Ordinary National Certificate or Diploma, or a course of equivalent standard, and normally have been in full-time or part-time attendance at an Establishment for Further Education for not less than two years immediately preceding 1st August, 1950. A recommendation from the Principal of the Establishment which the candidate has been attending must accompany all applications.

The award of a Technical State Scholarship will not exempt the holder from the normal admission requirement of a University. Successful candidates must make their own arrangements for acceptance by a University, University College, or Technical College. Candidates are strongly advised to secure provisional acceptance by the institution of their choice, making it clear that they are candidates for a Technical State Scholarship.

Explanatory leaflets and application forms may be obtained from Principals of Further Education Establishments or from the Ministry of Education, Curzon Street, London, W.1. Applications must be submitted through Principals to reach the Ministry not later than 30th June, 1950.

### Beilby Memorial Awards

The Administrators of the Sir George Beilby Memorial Fund, representing the Institute of Metals, the Royal Institute of Chemistry, and the Society of Chemical Industry, have decided to make three awards, each of one hundred guineas, from the Fund for 1949. These awards have been made to:

F. R. N. Nabarro, M.B.E., M.A., B.Sc.(Oxon.), in recognition of his application of mathematical methods to the elucidation of the mechanical properties of metals.

Charles Eric Ransley, M.Sc., Ph.D.(Lond.), F.I.M., in recognition of his experimental contributions to the knowledge of the behaviour of gases in metals.

Keble Watson Sykes, M.A., B.Sc., D.Phil.(Oxon.), in recognition of his experimental contributions to the study of the combustion of carbon and its oxidation by steam.

Awards from the Fund are made to British investigators in science as a mark of appreciation of distinguished work, particularly in such fields as fuel economy, chemical engineering, and metallurgy in which Sir George Beilby's special interests lay. In general, the awards are not applicable to the more senior investigators but are granted as an encouragement to relatively young men who have done independent work of exceptional merit over a period of years.

### Austrian Iron and Steel Institute

The Eisenhütte Öesterreich was formally constituted at a meeting held at the Montanistische Hochschule, Leoben, on 3rd February, 1950. The Institute will operate on lines similar to those of the Verein Deutscher Eisenhüttenleute.

In connection with the General Meeting of the new Institute, to be held from 15th–20th June, 1950, the Montanistische Hochschule has proposed to convene a conference on the Metallurgy of Welding.

### Norwegian Industries Development Association

The Norwegian Industries Development Association, Studieselskapet for Norsk Industri, has been founded as a private association open for membership to all Norwegian industrial or industrially interested firms and persons. The chairman is C. W. Eger, Managing Director of Elektrokemisk A/S, Oslo. It aims at investigating and developing possibilities of starting up new Norwegian industries, and where necessary, bringing up to date existing industrial activities.

The Association was created in secrecy, during the German occupation in 1943, by some of the leading industrialists of the country and came forward publicly shortly after the liberation.

The N.I.D.A. is one of the founders of the Norwegian Industries Commission set up in New York for the purpose of furthering contact between American and Norwegian industry and carrying out assignments for its members.

### Chatelier Centenary Celebrations

The Société Française de Métallurgie is organizing Centenary Celebrations of the birth of Henri le Chatelier, at a meeting to be held on 18th October, 1950. Dr. C. H. Desch, F.R.S., Past-President, has agreed to represent the Institute at the meeting.

### Summer School in Electron Microscopy, 1950

By the courtesy of Professor Sir Lawrence Bragg, O.B.E., F.R.S., a Summer School in Electron Microscopy will be held again this year in the Cavendish Laboratory, Cambridge.

The School will be held from 18th–29th July inclusive. A detailed syllabus and form of application for admission may be obtained from G. F. Hickson, M.A., Secretary of the Board of Extra-Mural Studies, Stuart House, Cambridge, to whom the completed application form should be returned not later than 5th June, 1950.

### Spectrography

The 13th Congress of the Groupement pour l'Avancement des Méthodes Spectrographiques will be held from 21st–23rd June, 1950, at the Laboratoire Central de l'Armement, 1 Place St. Thomas d'Aquin, Paris. The Congress will include four technical sessions, and, on the second day, a visit to a works or laboratory.

### Fourth World Power Conference

The Fourth World Power Conference will be held in London from Monday, 10th July, to Saturday, 15th July, 1950. The theme of the Conference is "World Energy Resources and the Production of Power." This

will be the first Plenary Meeting since the Conference held in 1936, in Washington, U.S.A.

In addition to a full technical programme, the Conference will include a number of tours in the United Kingdom, with visits to places of technical, historical, and scenic interest.

More detailed particulars may be obtained from The Conference Offices, Fourth World Power Conference, 414 Cecil Chambers, 76–86 Strand, London, W.C.2.

**Institution of Production Engineers**

The first Annual Summer School of Production Engineering is to be held at St. Peter's Hall, Oxford, from 30th August to 3rd September, 1950.

The Institution is establishing the Summer School to provide facilities for an interchange of views on specific aspects of production engineering, between those responsible for education and training and those in industry responsible for production. One of the main objects of the School is to foster the development of teaching methods. The theme for the 1950 School will be "The Science and Practice of Engineering Applied to Production."

Further particulars and application forms may be obtained from the Institution of Production Engineers, 36 Portman Square, London, W.1.

**Advisory Council of the D.S.I.R.**

The Lord President of the Council has appointed Professor Sir Ian Heilbron, D.S.O., F.R.S., to be Chairman of the Advisory Council for Scientific and Industrial Research in the place of Sir Geoffrey Heyworth who resigned owing to pressure of other public duties.

Sir Ian is now Director of Research of the Brewing Industry Research Foundation, near Redhill, Surrey.

**Operational Research**

Commencing in March, 1950, the Operational Research Club have issued a Quarterly Bulletin, concerned with the application of the scientific method to human behaviour, both by itself and in relation to the use of materials and equipment. Copies may be obtained at 10*s.* yearly (post free) from the Business Editor, 25 Buckingham Gate, London, S.W.1.

**Roll-Turning Lathe**

A new automatic roll-turning lathe, developed by the Monarch Machine Tool Co., of Sidney, Ohio, U.S.A., is claimed to be a great advance on conventional methods of contour roll turning. For example, a chilled iron contour roll 84 in. long × 18 in. dia. was turned in 4½ hr. in place of the previous time of 7 days. Fitted with Keller magnetic control the machine is stated to be exceptionally robust, sensitive, and accurate. The agents in this country are the Rockwell Machine Tool Co., of Wembley, Middlesex.

**Precision Metal Forming**

The Glenn L. Martin Co., U.S.A., have recently developed the Marform Process, a new, efficient, and economic method of precision metal forming. In the United Kingdom, the British Commonwealth, Europe, and other export territories the Loewy Engineering Co., Ltd., will supply, by arrangement with the American Company, complete presses or equipment for adapting existing presses to the new process.

**Land Pyrometers, Ltd.**

A new Company has been formed to acquire and carry on the business in pyrometric equipment which has hitherto been conducted by T. Land and Son, Ltd. The name and address of the new Company is: Land Pyrometers, Ltd., Colonial Works, Queen's Road, Sheffield, 2. Telephone: 20613 and 20809. Telegraphic address: Pyrometers Sheffield 2.

**William Jessop and Sons, Ltd.**

The London Office of Messrs. William Jessop and Sons, Ltd., and J. J. Saville and Sons, of Sheffield, is now Kingsway Chambers, 44–46 Kingsway, London, W.C.2. Telephone: Holborn 7145/6.

**Unionmelt Welding Process**

As from 1st April, 1950, all sales and service for Unionmelt Automatic Arc Welding equipment in the United Kingdom is being handled by the Quasi-Arc Company, Ltd., Bilston, Staffs.

## CORRIGENDUM

**First Report of the Ingot Surface Defects Sub-Committee**

A sentence was omitted from the MS of this report, which was published in the May issue of the *Journal*. The sentence reads: "The surface at positions between the cracks was neither unduly decarburized nor coarse-grained," and should be inserted on p. 53, in the fourth line up from Fig. 17, after the words ". . . coarse-grained."

## DIARY

**28th May–6th June**—THIRD WORLD PETROLEUM CONGRESS—The Hague, Holland.

**21st–23rd June**—13TH CONGRESS, GROUPEMENT POUR L'AVANCEMENT DES METHODES SPECTROGRAPHIQUES—Laboratoire Central de l'Armement, 1 Place St. Thomas d'Aquin, Paris.

**26th June**—AMERICAN SOCIETY FOR TESTING MATERIALS—53rd Annual Meeting—Atlantic City, U.S.A.

**28th June**—IRON AND STEEL ENGINEERS GROUP—Thirteenth Meeting—4 Grosvenor Gardens, London, S.W.1, 10.0 A.M.

**29th June–1st July**—INSTITUTE OF PHYSICS (Manchester and District Branch)—Summer Conference. "*The Physics of Lubrication*,"—The University, Manchester.

## TRANSLATION SERVICE

(The previous announcement was made in the May, 1950, issue of the *Journal*, p. 102).

TRANSLATIONS AVAILABLE

No. 398 (German). W. OELSEN: "The Thermodynamics of Iron Alloys." (*Stahl und Eisen*, 1949, vol. 69, July 7, pp. 468–474; discussion, pp. 474–475).

No. 399 (German). H. EULER: "International Comparison of Production, Personnel and Efficiency in the Iron and Steel Industry." (*Stahl und Eisen*, 1949, vol. 69, Nov. 10, pp. 827–835).

TRANSLATION IN COURSE OF PREPARATION

(German). M. HANSEN: "Fundamentals of Flow in Industrial Furnaces." (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 337–344).

CHARGES FOR COPIES OF TRANSLATIONS—The charge for the above translations is £1 for the first copy and 10*s.* for each additional copy of the same translation. Requests should be accompanied by a remittance. These translations are not available on loan from the Joint Library.

TRANSLATIONS PREPARED AT MEMBERS' REQUEST—Members requiring translations of foreign papers are invited to communicate with the Secretary, who will ascertain whether they can be prepared for inclusion in the Series.

# ABSTRACTS OF CURRENT LITERATURE *and* BOOK NOTICES

IRON AND STEEL MANUFACTURE AND RELATED SUBJECTS

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## FUEL—PREPARATION, PROPERTIES, AND USES

**The Design of Contra-Flow Heat Exchangers.** E. Schmidt. (Proceedings of the Institution of Mechanical Engineers, 1948, vol. 159, War Emergency Issue No. 44, pp. 351–362). The problem of contraflow heat exchangers, for gases at turbulent flow, is treated in a general way, using the analogy between heat transfer and friction. To make this analogy more evident, another non-dimensional heat-transfer coefficient is introduced. This number may be used profitably instead of the Nusselt number for describing heat transfer in tubes, and in similar problems. Thus the main dimensions of the apparatus are given in formulæ based on the intended heat exchange, the total drop in temperature, the total loss of energy, and the properties of the fluid. The weight and total volume of the heat exchanger depend on the ratio of temperature drop and loss of energy on both sides of the heat exchanging surface. The conditions of minimum weight and minimum volume are derived.—R. A. R.

**The Coking Industry, with Special Reference to Great Britain.** D. Hicks and G. W. Lee. (United Nations Scientific Conference, Fuels and Energy Section : Coke and Gas, 1949, vol. 11, Oct., pp. 361–366). This survey of the coking industry covers the range of properties of the coals in Great Britain, the reserves and production of coking coals, factors affecting coke quality, developments in plant design, coke quenching, and by-product recovery.—R. A. R.

**The Quality of Coke for Blast Furnaces.** T. Sanderson. (Coke Oven Managers' Association, Gas World, 1949, vol. 130, Oct. 1, Coking Section pp. 7–10). The value of the shatter test to the blast-furnace operator as an indication of coke quality and its performance in the furnace is discussed and the need for another test is shown. The results of a statistical analysis of data on coke consumption per ton of pig iron indicated that the $-\frac{1}{2}$-in. shatter index had the most significant correlation and that most of the information given by the 2-in. and $1\frac{1}{2}$-in. shatter indices was given by the $-\frac{1}{2}$-in. index alone.—R. A. R.

**Physico-Chemical Aspects of the Carbonisation of Coal.** J. J. Kipling. (Science Progress, 1949, vol. 37, Oct., pp. 657–669). Research work on the carbonization of coal is cited and the following sequence of events with increasing temperature is deduced : (1) Removal of water, carbon dioxide, and other occluded gases ; (2) removal of waxes and resins by simple distillation ; (3) simultaneous breakdown of the main coal matter, with production of distillate and condensation of the remaining solid matter into layers of hexagonal rings which become progressively more aromatic in character ; (4) loss of radicals bound to the edges of the layer planes ; and (5) loss of the residual hydrogen and oxygen from the borders of the planes as water gas, leaving the hexagons progressively more unsaturated and more graphitic ; the $H_2/CO$ ratio increases with rising temperature and is highest for high-rank coals. The physical changes accompanying these chemical changes and the effects on the physical properties of the coke are discussed. There are 30 references.

R. A. R.

**The Measurement of Coke Yield at Coking Plants.** (British Coke Research Association, Oct., 1949, Panel No. 2, First Report to the Research Committee). Where accurate weighing facilities are not available some inferential method of determining the coke/coal weight ratio is essential. Such methods have been investigated and the ash-ratio method found unsatisfactory. That based on the volatile matter content of the coal and coke can be used, but it is subject to considerable error if the sampling and analysis are not carried out with a high degree of accuracy.—R. A. R.

**The Production, Properties and Use of Coke Breeze from Coking Plants.** (British Coke Research Association, May, 1949, Panel No. 3, Second Report to the Research Committee). Information on coke breeze obtained by a questionnaire circulated to all coking plants in Great Britain is analysed, the properties of the breeze are described, and possible lines of research work on the development of new outlets for this material are indicated.—R. A. R.

**An Application of the Fluidized Solids Technique to Coal Gas Purification.** R. H. Williamson and J. E. Garside. (Institution of Gas Engineers, Nov., 1949, Communication No. 357). Laboratory tests are described in which hydrogen sulphide was removed from coal gas by means of a fluidized bed of powdered iron oxide.—R. A. R.

**Modern Metering Instruments for Determining Gas Quantities in Metallurgical Works and Blast-Furnace Plants.** J. Kesper. (Metallwirtschaft, 1949, vol. 3, Oct., pp. 332–333). A brief description is given of low-pressure and high-pressure balanced ring flowmeters for measuring the flow of gases in pipelines. This meter consists essentially of a ring of metal tube sealed internally by a disc on each side of which is a space closed by a liquid occupying slightly less than half of the tube. The ring is in a vertical plane and is supported and balanced on a knife edge at the centre. A pair of tubes connects the high and low pressure sides of an orifice plate in the gas main to the two spaces in the tube. Fluctuations in the pressure difference displace the sealing liquid, causing the tube to move so that a pointer attached to its periphery indicates the flow by moving across a calibrated scale.

R. A. R.

## TEMPERATURE MEASUREMENT AND CONTROL

**"Heat Inertia" in Problems of Automatic Control of Temperature.** V. Broida. (Instruments, 1949, No. 2, pp. 136–138, 160–164; No. 3, pp. 222–224, 254, 256, 258, 260, 262, 264; No. 4, pp. 324–325, 362, 364, 366; No. 5, pp. 406, 450, 452, 454, 456, 458: Chemical Abstracts, 1949, vol. 43, Aug. 10, col. 5648). The general form of the equation for variation of temperature inside a heating unit is developed, from which expressions are derived for the determination of the fictitious mass of the unit and the stabilization time required under new conditions. Theoretical and actual behaviours of all types of discontinuous and continuous automatic temperature-control systems are discussed. Equations are developed for each type relating fictitious mass with variations of heated-medium flow and resulting temperature changes, and the use of the equations in the selection of controllers for specific applications is shown.

**Pyrometry and Temperature Control.** M. W. Johns. (Canadian Metals and Metallurgical Industries, 1949, vol. 12, Nov. pp. 20–23, 42, 43). The principles of liquid and bimetal thermometers, pyrometric cones, and radiation and thermocouple pyrometers are explained with recommendations on the selection and maintenance of temperature measuring and control apparatus.—R. A. R.

**Instruments and Techniques in Industrial Heat Transfer Research.** R. H. Baulk. (Monthly Bulletin of the British Coal Utilisation Research Association, 1949, vol. 13, Nov., pp. 381–387). The author reviews the work of previous investigators in the field of heat transfer in combustion chambers and discusses means of measuring heat transfer, flame emissivity and temperature, and the sensible heat of combustion gases.—R. F. F.

**Investigations on the Measurement of the Temperature of Liquid Steel by Means of the Platinum/Platinum–Rhodium Thermocouple.** P. Bastien and J. Bleton. (Société Française de Métallurgie, Oct. 22, 1948: Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 807–817). The construction and use of the Schofield-Grace quick-immersion thermocouple and its precision in comparison with the disappearing-filament pyrometer are described, and some account of investigations of the distribution of temperature in the steel bath and its consequences is given.—A. E. C.

**The Tungsten–Iridium Thermocouple for Very High Temperatures.** W. C. Troy and G. Steven. (American Society for Metals, Oct., 1949, Preprint No. 19). Operating between 1600° and 2000° C., calibrations were made for thermocouples representing combinations of tungsten, molybdenum, tantalum, platinum, rhodium, iridium, and alloys of these metals. The tungsten–iridium thermocouple appeared to possess the most desirable combination of properties for use in the temperature region 1000° to 2100° C. The thermoelectric characteristic curve was almost straight; the e.m.f. output was high and remained constant in helium for 120 hr. Only inert gases or vacuum were suitable for operation of the thermocouple because of the embrittlement of iridium in hydrogen and the limitations with the use of tungsten from the point of view of the ambient atmosphere.—J. C. R.

**Installations and Maintenance of Pyrometers in Hardening Shops.** G. Oestereich. (Werkstatt und Betrieb, 1949, vol. 82, Oct., pp. 367–369). Recommendations are made on the fitting of thermocouples in heat-treatment furnaces and the calibration and maintenance of circuits and instruments.

R. A. R.

## REFRACTORY MATERIALS

**Refractories for the Iron and Steel Industry. Progress over a Quarter Century.** A. E. Dodd. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 220–225, 237). Developments during the past 25 years in refractory materials used in blast-furnaces, open-hearth furnaces, and casting pits, are reviewed.—J. C. R.

**Heat Transfer in Refractory Insulating Materials. Part II. Studies of a Heat Dissipation Test for Insulating Materials.** L. R. Barrett, J. Vyse, and A. T. Green. (British Ceramic Research Association: Transactions of the British Ceramic Society, 1950, vol. 49, Mar., pp. 95–121). A study has been made of a simple test which compares the insulating power of bricks on the basis of the watts consumed by an electric heater placed inside a bored out prism of standard size cut from the brick, when the internal temperature is maintained 900° C. above room temperature. The many factors of an accidental kind or associated with the brick which may influence the heat dissipated have been investigated. Inadequate sampling, a departure from the prescribed bore diameter, a change of heater size and exceptionally low emissivities are the factors most likely to cause appreciable deviations from equivalence with the more trustworthy full-scale thermal conductivity test. Such virtual equivalence is established by comparing the watts dissipated from test-pieces cut from 13 brands of brick with the thermal conductivity by the calorimetric slab test method. The latter was measured perpendicular to the 9 × 4½-in. and 9 × 3-in. faces and averaged. It is suggested that a second standard temperature interval of 1300° C. would prove useful for bricks suitable for use at about 1300° C.

**Heat Transfer in Refractory Insulating Materials. Part III. Thermal Conductivity and Permeability as Related Directional Properties.** L. R. Barrett, J. Vyse, and A. T. Green. (British Ceramic Research Association: Transactions of the British Ceramic Society, 1950, vol. 49, Mar., pp. 122–128). From measurements of thermal conductivity and air permeability of 13 insulating bricks, perpendicular to both the 9 × 4½-in. and 9 × 3½-in. faces it is concluded that these are inter-related. Some hitherto unobserved pressure differences in the hot and cold faces of the test slab in the thermal conductivity test apparatus are tentatively ascribed to the phenomenon of thermal transpiration, which is known to be significant in fine-pored materials.

**Notch Sensitivity and Low-Temperature Spalling of Fireclay Refractories.** R. Rose and R. S. Bradley. (Journal of the American Ceramic Society, 1949, vol. 32, Nov., pp. 360–361). Attempts were made to correlate the physical properties of fireclay refractories with the phenomenon of low-temperature spalling. A method of considerable promise was developed in which the relative flexural strength of fireclay refractories at 1500° F., in the notched and unnotched conditions indicated the resistance of these bodies to low-temperature spalling. No purely mathematical relationship between the physical properties and low-temperature spalling characteristics appeared to be valid.

**Modern Refractories Give New Operating Economies.** R. P. Heuer and M. A. Fay. (Iron Age, 1949, vol. 164, Oct. 6, pp. 82–85; Oct. 13, pp. 86–90). The authors discuss the use of carbon refractories in the blast-furnace; early promise has not yet been fulfilled, as carbon hearths are not, as was supposed, immune from 'breakouts' and the formation of salamander. The use of monolithic sillimanite as a large pad under the hearth does, however, tend to restrict salamander accumulations, and this material can also be used for ramming between carbon blocks and the hearth staves. In the hearth and bosh sections of the furnace it seems to justify its extra cost. They next consider 'high-fired' brick which is resistant to carbon monoxide disintegration as all the iron oxide is converted to silicate by the firing: this material is of great use in the stack lining, and in blast-furnace stoves; it resists alkali penetration, a severe risk in the latter application. In the second part of this article, the authors consider open-hearth refractories, describing the development of chrome-magnesite steel-cased brick, and of special silica-refractory brick. In mixers, both chrome-magnesite and sillimanite brick are used, and in mixers at lower temperatures, high-duty fireclay bricks have proved satisfactory. Economy, the authors point out in conclusion, depends on a careful study of all the factors involved.—J. P. S.

**Use of Grog Nozzles in Open-Hearth Furnaces.** N. F. Dubrov and M. D. Nikitenko. (Ogneupory, 1948, vol. 13, pp. 560–563: Chemical Abstracts, 1949, vol. 43, Aug. 10, col. 5916). The substitution of grog for Dinas brick increased the life 18%. Greater improvement can be obtained by decreasing the porosity of the brick. Brick was made from Chasov Yar clay 25%, Vogul'sk clay 25%, and grog from Vogul'sk clay 50%. Brick analysed $SiO_2$ 57·8%, $Al_2O_3 + TiO_2$ 39·98%, $Fe_2O_3$ 0·78%, CaO 0·42%, MgO 0·61%, and ignition loss 0·14%; refractoriness 1720°; porosity 22·7%; mechanical strength 287 kg./sq. cm.; additional strength at 1350°, 1·15%; and initial softening under 2 kg./sq. cm. at 1390° C.

**Determination of the Resistance to Erosion and Slag Attack of Steel Ladle Bricks.** E. Rousseau. (Chimie et Industrie, 1949, vol. 62, Sept., pp. 243–248; Oct., pp. 362–370). A method of determining the resistance of ladle bricks to molten

steel and slag is described. It consists of plunging simultaneously prismatic specimens, cut from the bricks and from bricks of known life, into a bath of molten steel or of fluid slag of suitable basicity brought to the casting temperature, and holding them there for a suitable time. With silica brick, slag penetrates deeply into the mass; firing silica bricks at 1420–1450° C. results in a partially fritted material, dense, with a greater refractoriness-under-load, and more resistant to attack than bricks fired at a lower temperature. Good ladle bricks should contain more than 38% of alumina, the volume porosity should be 20–22%, have a good cold crushing strength, an initial softening temperature above 1300° C., and a refractoriness greater than 1700° C. Bricks with high grog content can fulfil these conditions provided they have been fired in the 1420–1450° C. range.

An English translation of this paper has been made (*see* Iron and Steel Institute Translation Series No. 396).—R. A. R.

**Cupola Refractories.** W. A. Archibald. (Iron and Steel, 1949, vol. 22, Nov. 22, pp. 515–518). A review is presented of the literature dealing with cupola refractories, and in particular the requirements of the melting zone. The work of various investigators on firebricks and silica bricks, monolithic acid lining material, acid patching materials, basic lining materials, cupola design and construction, operating conditions and lining life, and slag mechanism, is reported, and general conclusions are drawn.—J. C. R.

## BLAST-FURNACE PLANT AND THE PRODUCTION OF PIG IRON

**Ironmaking.** H. L. Saunders. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 132–134). Developments in ironmaking practice during the past 25 years are discussed.—J. C. R.

**Blast Furnace Has All-Welded Earthquake-Proof Substructure.** (Industrial Heating, 1949, vol. 16, Nov., pp. 2046–2047). An all-welded blast-furnace, designed to resist earthquake shocks at the steelplant of Compania de Acero del Pacifico, Bay of Vincente, Chili, is briefly described. The base, columns, and mantel are made up of rectangular, hollow, box-girder sections, fabricated by welding, from 1¼-in. steel plates. For this furnace with a 20-ft. hearth and 12 tuyeres there will be 8 instead of the conventional 12 columns. There will be a great saving in weight of material.—R. A. R.

**Pressure Operation. Modern Trends in Blast Furnace Practice.** J. M. Ridgion. (Iron and Steel, 1949, vol. 22, Oct. pp. 443–447). After tracing the history of blast-furnace pressure operation, the author outlines the theory of this method of working and discusses furnace modifications that are required. Operating data from pressure and normal blast-furnace operations are compared, allowance being made for some varying factors.—J. C. R.

**The Reduction of Iron Oxides in Melts.** E. J. Kohlmeyer. (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 345–347). As a result of observations during the process of determining oxygen in powders of iron, steel, and other metals, investigations were carried out on the reduction of iron oxide and mixtures of iron oxides and other oxides by melting in a carbon crucible. At a given temperature the rate of reduction is dependent on the heats of formation of the added oxides, their melting points, and the solubilities of the reduced metals in molten iron. The vaporization which occurs during the reduction of iron oxide in a melt is caused primarily by the formation of an unstable Fe–CO complex which occurs at 1400° C. This complex must play an important part in the formation of dust in the blast-furnace and in the attack on open-hearth furnace roofs.—R. A. R.

**Relative Reducibility of Some Iron Oxide Materials.** E. P. Barrett and C. E. Wood. (United States Bureau of Mines, Oct., 1949, Report of Investigations No. 4569). A standard procedure has been developed by which the relative reducibility of iron oxide materials in hydrogen, in carbon monoxide, and mixtures of these gases, could be rapidly and accurately determined. The apparatus and procedure are described and the results obtained discussed. It was established, among other things, that the descending order of reducibility by hydrogen at 900° C. of six groups of materials is (1) limonite, (2) hematite, (3) pellets formed by balling fine moist oxide in a rotating drum at room temperature and then heating to develop a bond, (4) sinter, (5) nodules formed by heating the oxide to incipient fusion in a rotary kiln, and (6) magnetite.

## PRODUCTION OF STEEL

**The Steel Development Plan in Action.** (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 42–131). Detailed reports are presented of modernization and expansion of plant of the following companies and their associated companies: Colvilles Ltd.; John Baker and Bessemer Ltd.; Consett Iron Co., Ltd.; Darlington and Simpson Rolling Mills, Ltd.; Dorman Long and Co., Ltd.; English Steel Corporation, Ltd.; Firth-Vickers Stainless Steels, Ltd.; Samuel Fox and Co. Ltd.; Guest, Keen and Nettlefolds (South Wales), Ltd., Hadfields, Ltd.; Richard Johnson and Nephew, Ltd.; The Lancashire Steel Corporation, Ltd.; Arthur Lee and Sons, Ltd.; John Lysaght's Scunthorpe Works, Ltd.; The Park Gate Iron and Steel Co., Ltd.; Round Oak Steel Works, Ltd.; Skinningrove Iron Co., Ltd.; South Durham Steel and Iron Co., Ltd.; The Steel Company of Wales, Ltd.; Stewarts and Lloyds, Ltd.; John Summers and Sons, Ltd.; Richard Thomas and Baldwins, Ltd.; The United Steel Companies, Ltd.—J. C. R.

**Twenty Years of Steelmaking.** T. P. Colclough. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 135–137, 134). Developments in steelmaking practice during the period 1928–1948 are reviewed.—J. C. R.

**The Modern Production of Iron and Steel.** G. R. Bashforth. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 138–148, 161). An illustrated account is given of iron and steelmaking processes from earliest times to the present day.—J. C. R.

**Trends in Iron and Steel Manufacture.** Sir Andrew McCance. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 185–188, 203). Improvements in iron and steelmaking techniques over the past 20 years are broadly considered.—J. C. R.

**How Timken Expedites Steel Mill Scrap Preparation.** F. C. Wier. (Iron Age, 1949, vol. 164, Sept. 15, pp. 88–90). At Jambrinus, Ohio, the Timken Roller Bearing Co. maintains a scrap yard where incoming material, *e.g.*, open-hearth 'pan cakes,' and Bailey bridge scrap, is cut to size by oxy-acetylene or the Lende powder-cutting process. Oxygen is supplied to cutting stations by a pipe network; both cylinder and generator acetylene are available.—J. P. S.

**New Steel Plant at Port Talbot.** (Overseas Engineer, 1949, vol. 23, Nov., pp. 131–135). An illustrated account is given of the design and present state of erection of the ingot-mould-preparation building at the new steelworks at Port Talbot. R. A. R.

**Steel, Peech & Tozer—An Account of the Templeborough and Ickles Steel Works.** (United Steel Companies, Ltd., 1949). This 64-page book contains the history and development of Steel, Peech & Tozer, and profusely illustrated descriptions of the steelmaking, rolling, forging, and finishing processes at their Sheffield works. Internal administration, education, training, and welfare are also dealt with.—R. A. R.

**Some Practical Aspects of Recent Developments in Open Hearth Furnace Design and Operation.** A. H. Leckie. (West of Scotland Iron and Steel Institute, Nov., 1949, Advance Copy). The author reviews the results of research and developments in open-hearth furnace design and operation, and their effect on three main groups of problems concerned with (1) the flues, regenerators, and uptakes, (2) the ports, and (3) the melting chamber. During the last few years the main trend has been towards greater simplicity of furnace design, but more complications in instrumentation and control. Removing carbon from the bath by blowing oxygen into it has reduced the decarburizing period, but many investigators say that compressed air is just as effective because it is the stirring action which is the real cause of the faster reaction. R. A. R.

**The Fundamentals of Flow in Industrial Furnaces.** M. Hansen. (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 337–344). After dealing with the laws of flow relating to continuity, energy, and impulse, the author discusses Reynolds numbers, laminar and turbulent conditions, the Prandtl friction layer, and flow in pipes with rough and smooth walls and with changes in section and direction. Wall turbulence and free turbulence are given special attention and examples of them are described. Secondary flow, the determination of furnace pressure, and the importance of the use of models are also considered.

**Scrap Charging—Key to Increased Open-Hearth Production.** R. R. Fayles. (American Institute of Mining and Metallurgical Engineers, 1949, Open-Hearth Conference Proceedings, vol. 32, pp. 23–34). *See* Journ. I. and S.I., 1950, vol. 164, Apr., p. 486.

**Effect of High Coke-Oven Gas Firing on Open-Hearth Operation.** F. G. Jaicks. (American Institute of Mining and Metallurgical Engineers, 1949, Open-Hearth Conference Proceedings, vol. 32, pp. 35–41): Blast Furnace and Steel Plant, 1949, vol. 37, July, pp. 808–812). A study has been made of the effects of greatly increasing the proportion of coke-oven gas in the firing of open-hearth furnaces with oil and gas combined. There was a definite limit to which the oil input could be decreased below which the rate of heat transfer declined. The optimum production of steel and fuel economy were obtained by using coke-oven gas at a rate which provided 30% of the total B.Th.U. input.—R. A. R.

**Sulphur in Basic Iron and Steel.** H. E. Warren, jun. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 55–65). Data relating to changes in the sulphur content of blast-furnace iron and at various stages in the production of steel have been collected and studied. The following points were noted: (1) With increasing sulphur in the iron, the decrease in sulphur in the ladle became greater; (2) high-sulphur iron was not produced when the manganese content was over 2·80%; (3) there was no relation between the sulphur in the iron and that in the steel either in the furnace or in the ladle; (4) the drop in the sulphur in the iron transfer ladle was about 0·20% greater when the manganese was above 2·80% than it was with less than 2·80% of manganese; (5) the sulphur content at the time of casting pigs rose directly with the temperature of the iron; (6) at the pig-casting machine the product % Mn × % S increased directly with temperature; (7) when the manganese in the iron as-cast was over 2·80%, the sulphur was in every case below 0·065%; and (8) the sulphur content of the steel on tapping varied with the sulphur content of the fuel.—R. A. R.

**Effect of Charging Variables and Additions on Sulphur Elimination.** A. H. Osborne. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 66–71). Data on variables affecting the sulphur content of 130 heats of open-hearth furnace steel have been collected and subjected to statistical analysis. The two most important factors affecting the sulphur in the steel were found to be the sulphur in the materials charged and the basicity of the slag. Other significant points were: (1) The greater the amount of rusty scrap charged the higher was the sulphur; (2) increasing the works-scrap/bundled-scrap ratio reduced the sulphur in the steel; (3) longer charging times caused higher sulphur in the steel; and (4) increasing the proportion of sinter in the charge tended to lower the sulphur content of the steel.—R. A. R.

**Use of Manganese Ore for Sulphur Removal.** W. H. Carpenter. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 71–72). Factors affecting the removal of sulphur in open-hearth furnace practice have been examined by the Colorado Fuel and Iron Corporation. If the metal charged contained 0·80 to 1·20% of silicon, the consumption of lime need not be increased. Increasing the lime/silica ratio above 3·5–4·0 did not improve the removal of sulphur. Additions of 1000 to 3000 lb. of manganese ore (Mn 56%, Fe 4·00%, $SiO_2$ 6·8%, P 0·07% with no sulphur) successfully removed the sulphur from 115-ton heats.—R. A. R.

**Sulphur Balance in Open-Hearth Heats.** P. Schane, jun. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 72–74). Sulphur balances are being prepared at several works of the Carnegie Illinois Steel Corporation, and some preliminary findings are reported. With 65% hot-metal charges, the percentage of sulphur in the steel is made up of the following contributions: from the iron 0·020–0·030%, the scrap 0·009–0·021%, the limestone 0·005–0·009%, and the ore 0·003–0·004%. As to the fuels, tar mixed with coke-oven gas contributes 0·009 to 0·030% on heavy-scrap charges, and up to 0·050% on lighter-scrap charges. The run-off slag removes 10–30% of all the sulphur in the system, and the slag at tapping 14 to 45%.—R. A. R.

**Reports by Sections on Use of Oxygen and Compressed Air.** J. S. Marsh, E. H. Reyer, G. C. Lawton, T. A. Cleary, R. S. Bower, A. W. Thornton, and G. B. McMeans. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 75–87). Each of the above authors reports on the manner in which oxygen or compressed air is used for steelmaking, and in some cases on the results obtained, in the particular steelmaking district of the U.S.A. which he represents.—R. A. R.

**Factors Affecting Open-Hearth Production in a Gas-Fired Furnace.** S. F. Elam. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 87–93). Some results are reported of a statistical analysis of the effect of several variable factors on the tons of steel produced per hour in 150-ton open-hearth furnaces fired by coke-oven gas and natural gas. In general, the rate of production was increased by (*a*) increasing the weight of scrap and pig iron charged, (*b*) decreasing the proportion of cold pig iron, (*c*) decreasing the lime, (*d*) increasing the silicon content of the pig iron, (*e*) decreasing the time between tapping the blast-furnace and charging the molten iron, and (*f*) decreasing the charging time. Additions of from 2000 to 11,000 lb. of feed ore kept production at a high rate, but additions outside this range caused a rapid falling off in production.—R. A. R.

**Evaluation of Factors Influencing Open-Hearth Furnace Production in Oil-Fired Furnaces.** A. P. Woods. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 93–101). Statistical methods were employed to evaluate the effects of variables on the tons of steel produced per hour in basic open-hearth oil-fired furnaces. It was found that considerable expenditure on equipment to enable various operations, such as charging, to be speeded up would be necessary in order to obtain any substantial improvement in the production rate. The hot metal can be added too soon after finish charging, the optimum period being 40 to 60 min. A high sulphur content at 'melt-down' can be worked down without delay if the $SiO_2$/CaO ratio is high enough; it is the addition of burnt lime to correct a low $SiO_2$/CaO ratio which causes delay.—R. A. R.

**Open-Hearth Production Rates.** R. H. Ede. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 102–103). The results of statistical analyses of factors affecting production rates presented by S. F. Elam and by A. P. Woods (*see* preceding abstracts) are compared with those obtained by the Carnegie Illinois Steel Corporation. It was brought out that scrap containing copper absorbed more sulphur from the fuel than scrap with no copper content, and in consequence, when producing steel with 0·035% max. of sulphur, the heat time was considerably longer when the residual copper in the scrap was rather high (0·07%).—R. A. R.

**Sampling of Oxygen in Waste Gases.** G. C. Primm. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 104–106). This paper summarizes the answers to a questionnaire received from 42 steelworks regarding the practice of determining the oxygen in the waste gas from open-hearth furnaces.

**What Is Being Done to Improve Scrap Quality and Better Prepare It for Charging?** H. A. Parker. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 107–109). An analysis is made of the answers received from 51 open-hearth melting shops on the methods employed to prepare scrap and reduce it to charging-box size.—R. A. R.

**Use of Blown Metal in Stationary Furnaces.** S. J. Dougherty. (American Institute of Mining and Metallurgical Engineers, 1949, Open Hearth Conference Proceedings, vol. 32, pp. 110–115). Bessemer converter practice at the Weirton Steel Company's plant is described. Two 25-ton converters and one spare one are employed. The blast-furnace iron charged contains silicon 1·20–1·40%, manganese 1·00–1·15%, and phosphorus 0·360%. Mixed with about 12% steel scrap, it is blown down to 1·75% of carbon in about 9 min. Steam is used when the scrap is light or the iron high in silicon. The blown metal is then poured into an open-hearth furnace already charged with limestone and scrap. The plant uses 400-ton open-hearth furnaces with charges composed of 70% blown metal, 30% steel scrap, and 5% limestone. The average time for a heat (from tap to tap) is now 14 hr.

**Instrumental Control of Open Hearth Furnaces.** R. Toye and R. Haynes. (Instrument Practice, 1949, vol. 3, Aug., pp. 441–443; Sept., pp. 497–501; Oct., pp. 553–556). A brief account is given of what are believed to be the main variables affecting the operation of the open-hearth furnace and how they can be measured and controlled by the melter to assist him in producing the maximum output of good steel with a minimum consumption of fuel and refractories. In Part I these variables are discussed and the measurements that should be made to control combustion and heat transfer are indicated. In Part II the principles applied in making flow, pressure, and temperature measurements in a steelworks are explained and precautions to be taken in the installation and use of different instruments are discussed. In Part III the authors describe how instrument readings are being used in practice to determine the optimum conditions of operating the furnace in question, and to maintain these conditions over prolonged periods.—R. A. R.

**Some Notes on Open-Hearth Practice.** R. L. Knight. (Mechanical Engineers Association: Australasian Engineer, 1949, Nov. 7, pp. 90–95). The design and layout of a modern open-hearth melting shop are discussed and steelmaking practice at an Australian plant is described.—R. A. R.

**The Industrial Uses of Oxygen-Enriched Air.** P. Guillaumeron. (Chimie et Industrie, 1949, vol. 62, Oct., pp. 354–361). The industrial uses of oxygen are briefly reviewed with descriptions of how oxygen is injected into open-hearth and electric steel furnaces, and its application in water gas and ammonia production.—R. A. R.

**The Dephosphorization of Iron Alloys with Alkaline and Alkaline-Earth Metals, Preferably with Magnesium.** W. A. Fischer and J. Heyes. (Mitteilungen aus dem Max-Planck-Institut für Eisenforschung No. 503: Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 369–374). When phosphorus-rich iron alloys, or ferrophosphorus (P 22%), or ferromanganese (Mn 66%, P 2·6%) were heated with magnesium in a closed iron crucible, and the reaction products leached with water, most of the phosphorus was driven off as gaseous phosphoretted hydrogen. Even at 700–800° C. magnesium reacted with ferrophosphorus causing up to 95% of the phosphorus to be removed. In the case of the ferromanganese, an increase of about 100° C. was required to remove the same proportion of phosphorus. Tests in a rotary furnace with a protective atmosphere of hydrogen and much larger samples of 500 g. confirmed the previous results on 3 to 10-g. samples.—R. A. R.

**Induction Stirring in Electric Furnace Steel Making.** E. S. Kopecki. (Iron Age, 1949, vol. 164, Sept. 22, pp. 73–78). The author examines the advantages of induction stirring in the electric furnace as described by recent Swedish workers. The stirring unit evolved resembles a section of the stator of an alternator; it is installed directly under the bath, and operates by driving the melt diametrically across the furnace bottom. The metal then returns by semi-circular eddies to its original point of departure. The effects of this type of stirring are said to include: (1) The attainment of a low carbon content without increased oxygen content; (2) less iron loss in the slag; (3) more rapid removal of sulphur; and (4) greater homogeneity in carbon, manganese, and silicon.

J. P. S.

**American Practice in the Acid Electric Steelmaking Furnace.** A. C. Brearley. (Metal Treatment and Drop Forging, 1949–50, vol. 16, Winter Issue, pp. 253–258). The author gives an account of acid electric furnace steel-melting practice in the U.S.A. For all grades of steel great emphasis is placed on promoting a short brisk boil. On completion of melting, oxygen is injected to promote the reduction of carbon and a lively boil. Oxygen consumption is about 80–100 cu. ft./ton (2240 lb.) of steel; at the same time electricity consumption is reduced by from 30 to 60 kWh./ton as compared with ore practice. Electricity consumption varies between 550 and 650 kWh./ton and electrode consumption from 7 to 10 lb./ton of steel produced.—R. A. R.

**Suspensions and Intergranular Substances in Steel.** R. Schäfer. (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 349–353). It is assumed that a steel bath contains extremely finely divided oxide impurities in suspension and that, on freezing, these are trapped in the grain boundaries. An investigation of the origin, causes, and prevention of these inclusions is described. They can be revealed for examination under the microscope by etching in a very weak solution of iodine in alcohol. Their origin can frequently be traced to poor quality scrap, ferromanganese, ferrosilicon, and other ferro-alloys. Factors tending to prevent the occurrence of these inclusions are deoxidizing with aluminium, shallow baths, and electric-furnace rather than Bessemer or open-furnace practice. These minute inclusions reduce the notch-bar impact strength of the steel. Finally, the etching procedure is described.—R. A. R.

## FOUNDRY PRACTICE

**Fundamental Investigations with a Small Test Cupola.** E. Piwowarsky and K. Krämer. (Neue Giesserei, 1949, Sept., Supplement No. 1, pp. 3–10). The effects of coke size and blast quantity on combustion in a small cupola (12 in. in dia. and about 6 ft. 6 in. high) have been investigated. The following conclusions were reached: (1) The curves for temperature against height above tuyeres are shifted to higher temperatures by increasing the quantity of air, but the zone of maximum temperature remains at the same height. The height of a zone of given temperature, *e.g.*, 1600° C. is, however, increased. The maximum $CO_2$ content of the gases is not altered. (2) Increasing the size of the coke raises the level of the maximum temperature zone. (3) Increasing the size of the coke raises the zone of maximum $CO_2$ content. In spite of different temperature maxima, the highest $CO_2$ contents measured in this zone with equal quantities of air remain about the same at 17%. For each size of coke the maximum temperature and the maximum $CO_2$ content are at the same height above the tuyeres. Increasing the size of the coke lowers by increasing amounts the reduction above the zone of maximum $CO_2$ content. Reduction is less near the walls than at the centre, although there was no evidence that more air was flowing up the walls than in the middle. (4) Increasing the air temperature raises the rate of combustion of the coke and brings the zone of maximum temperature closer to the tuyere level; the peak temperatures attained are also increased. The maximum $CO_2$ contents are not related to the air temperature in the range 20° to 400° C. but remain constant at about 17%. The practical significance of these results is considered.—R. A. R.

**The Oxidation Losses in the Cupola and the Formation of Cupola Slags.** H. Jungbluth. (Neue Giesserei, 1949, Sept., Supplement No. 1, pp. 11–16). It is shown that the oxidation of iron, silicon, manganese, and carbon in the cupola is related to the composition of the waste gas and is not related to the prevailing quantity of blast. A method of calculating the composition of the slag from the oxidation loss, coke ash, lime content, adherent sand, and cupola lining material is explained, and it is shown that the slag composition is related to the general composition of the cupola gases. The amount of blast necessary for slag production is discussed and the author's test results are compared with those in the literature.

R. A. R.

**Adjusting Composition in the Cupola.** (Metallurgia, 1949, vol. 40, Oct., pp. 335–336). A plant has been set up in Sheffield for the manufacture of briquettes of ferrosilicon, ferromanganese, and ferrochromium. These are made up from carefully screened material with a refractory binder. Distinctive shapes and colour dyes are employed to prevent mistakes in making additions to the cupola. With the present shortage of high-silicon and hematite irons these briquettes provide a convenient method of controlling alloying additions to the cupola charge.—R. A. R.

**Explosions in the Cupola.** J. Léonard and E. Waroux. (Fonderie Belge, 1948, May–June, pp. 20–23).

**Development and Present Position of Malleable Cast Iron.** F. Roll. (Neue Giesserei, 1949, vol. 36, Nov., pp. 339–347). The production and micro- and macro-structures of white-heart and blackheart malleable cast iron are described, and data on the tensile and fatigue strengths, weldability, corrosion and wear resistance, and machinability are presented and discussed. Finally, some applications, in particular car and lorry brake drums, are described.—R. A. R.

**Nodular Cast Iron.** M. M. Hallett. (Ship and Boat Builder, 1949, vol. 2, Jan., pp. 233–241: [Abstract] Bulletin and Foundry Abstracts of the British Cast Iron Research Association, 1949, vol. 10, July, p. 23). Limitations and advantages of the cerium process, from experience in the foundries of Sheepbridge Stokes Centrifugal Castings Company, are discussed.

**Nodulizing Gray Iron with Dilute Magnesium Alloy.** (Iron Age, 1949, vol. 164, Sept. 29, p. 65). A dilute magnesium alloy intended for producing nodular graphite in iron castings contains 7% magnesium and 45% silicon, the rest being iron : it is produced by the Electro Metallurgical Division of the Union Carbide and Carbon Corporation. It acts very effectively and the magnesium recovery is 35–65% depending on the pouring temperature. A briquetted mixture of magnesium and silicon–zirconium is also very effective, though magnesium recovery is not so high.—J. P. S.

**Magnesium Treatment for Nodulizing Graphite in Cast Iron. Dow Metal Chemical Co. Magnesium Division.** (Dowmetal Bulletin, 1949, Jan., No. DM109, pp. 1–5 : [Abstract] Bulletin and Foundry Abstracts of the British Cast Iron Research Association, 1949, vol. 10, July, p. 21). The following tentative conclusions are given : (1) Alloys of magnesium with high nickel or copper content react less violently with molten iron than magnesium alone, or other magnesium alloys. (2) Magnesium or magnesium alloys should be added to a slag-free surface of molten iron and allowed to float on the surface ; addition to the ladle during filling may cause the iron to be blown out of the ladle. (3) Magnesium-treated iron should be poured promptly after treating. (4) For best results, total carbon should be 3·0 to 4·0%, silicon 2·0 to 3·0% and sulphur 0·04% max.

**Nodular Graphite in Cast Iron.** H. W. Lownie, jun. (Metals Review, 1949, vol. 22, Mar., pp. 5–8). A review is presented of the published literature on nodular graphite formation in cast iron. The cerium treatment, the magnesium treatment, and the method using a nickel–magnesium alloy are described. J. C. R.

**The Progressive Steps in the Study of Graphite Formation.** J. G. Pearce. (Neue Giesserei, 1949, Sept., Supplement No. 1, pp. 17–21). The author gives an account of the work of the British Cast Iron Research Association from 1929 to 1948 on the formation of graphite in cast iron.—R. A. R.

**Cast Iron with Spheroidal (Nodular) Graphite Formation.** E. Piwowarsky. (Neue Giesserei, 1949, Sept., Supplement No. 1, pp. 23–31). The literature on the development of the production of cast iron with nodular graphite is reviewed, and the theory of the author and A. Wittmoser governing the mechanism of the formation of this graphite is explained. The position regarding patent priorities is also examined. R. A. R.

**The Particle Size of the Carbon Precipitated as Graphite in Malleable Cast Iron.** A. Wittmoser and E. Piwowarsky. (Neue Giesserei, 1949, Sept., Supplement No. 1, pp. 33–36). The importance of the free carbon (graphitic and temper carbon) in iron–carbon alloys in relation to wear resistance is discussed and the literature on the two forms is reviewed. An alloy was melted and annealed to produce graphite in lamellar form ; the graphite was microscopically examined *in situ* and, after extraction as a powder, its structure compared with that of carbon in pig iron, pearlitic cast iron, and black-heart malleable cast iron ; it was found to have the same structure as that in pearlitic cast iron. The particle size of the carbon as determined by X-rays was found to be practically the same in the two cases. From this it is deduced that the lamellar carbon produced by the special annealing process is identical with an ordinary grey iron so that similar behaviour under conditions of wear may be expected.—R. A. R.

**Steel Castings—A Review of Factors Affecting Surface Finish.** P. R. Beeley. (Iron and Steel, 1949, vol. 22, Oct., pp. 451–454). The work of various investigators who have studied the causes of burning-on or sand adherence to steel castings, is summarized. The conclusion is drawn that metal penetration and metal–mould interface reactions are the two principal factors concerned.—J. C. R.

**Pattern Equipment for Production Moulding.** S. A. Horton. (Institute of British Foundrymen : Foundry Trade Journal, 1950, vol. 88, Jan. 5, pp. 3–10). The fundamentals of pattern construction are reviewed and practical applications are dealt with with special reference to quality production and high-accuracy moulding. Various aspects of core-blowing practice are also discussed in detail.—R. E.

**Visit to a Patternmaking Works.** (Foundry Trade Journal, 1949, vol. 87, Dec. 29, pp. 741–746). The layout and equipment of the patternmaking works of G. Perry and Sons, Leicester, are illustrated and described.—R. E.

**Pattern Jigging on Moulding Machines.** H. Haynes. (Foundry Trade Journal, 1949, vol. 87, Nov. 3, pp. 537–542). The authors discuss the factors to be taken into account in the choice of moulding machines of the pattern-jigging type and describes several designs.—R. E.

**The Use of Plaster in the Pattern Shop and Foundry.** A. Bell. (Proceedings of the Australian Institute of Foundrymen, 1944–1947, vol. 4, pp. 97–101).

**Gates and Risers as Applied to Steel Castings.** E. R. Morgan. (Journal of the Birmingham Metallurgical Society, 1949, vol. 29, Sept., pp. 153–172). Recommendations on gates and risers for steel castings are made. The ingate orifice must be smaller than the sprue so that, for a given desired rate of pouring, the ingate cross-sectional area should be made up by multiple ingates ; this will at the same time give a lower Reynolds number for each ingate. The economic and technical aspects of bottom gating and gating at the parting line are discussed ; best results are obtained with multiple bottom ingates in conjunction with blind risers, wherever economic considerations permit. The design and positioning of open and blind risers are also dealt with.—R. A. R.

**Phenomena in the Solidification, Contraction, and Feeding in the Making of Castings.** D. Iturrioz. (Metalurgia y Electricidad, 1949, vol. 13, Apr., pp. 22–26 ; May, pp. 19–21 ; June, pp. 43–45 ; July, pp. 22–25). [In Spanish].

**The Feeding of Castings.** R. W. Ruddle. (Proceedings of the Institute of Australian Foundrymen, 1944–1947, vol. 4, pp. 119–129). The liquid contraction, solidification shrinkage, and solid contraction of castings, and how these can be influenced by open and blind risers are discussed.—R. A. R.

**Gates and Risers—Removal by Abrasive Cut-Off and by Band Saws.** H. J. Chamberland. (Iron and Steel, 1949, vol. 22, Oct., pp. 459–460). The advantages of using the friction band saw for removing gates and risers and other superfluous material up to 1 in. thick from ferrous castings, are discussed. Particular reference is made to its use with stainless steel castings.—J. C. R.

**Calculation of the Dimensions of Inserts at the Bottom of Risers to Facilitate Knocking the Risers Off the Solidified Casting.** P. S. Jashin. (Hutnické Listy, 1949, vol. 4, Aug., pp. 264–266). [In Czech]. The author has developed a new formula for the calculation of the dimensions of inserts placed at the bottom of the riser to facilitate subsequent removal of the riser. The usefulness of the formula has been confirmed by practical tests on grey-iron castings weighing between 1·5 and 35 tons. The numerical values of the factors required for calculating the dimensions of such inserts are given for grey-iron, steel, and bronze castings.—E. G.

**The Hydro-Blast Method of Cleaning Castings.** S. Fisher. (Proceedings of the Institute of Australian Foundrymen, 1944–1947, vol. 4, pp. 11–23). A detailed account is given of the plant, equipment, and process for the Hydro-Blast cleaning of both iron and steel castings. High-pressure water (at 1200 lb./sq. in.) is supplied at 30 gal./min., by a pump requiring 30 h.p., to a gun which entrains sand, and the operator directs the jet at the casting on a table in an enclosed booth. The floor of the booth is a grating through which the sand falls on to a screen ; water and screened sand are pumped to a spreader flume, screen, classifier, and dewatering equipment for recovery of the sand to be used again. Estimates of cost for installing and operating the equipment in Australia are given.—R. A. R.

**Skilful Moulding and Casting and Their Effects on the Price Structure of Castings.** J. H. Küster. (Neue Giesserei, 1949, vol. 36, Sept., pp. 260–264). Examples of moulding techniques for the economic production of grey-iron castings include several types of bearing supports, a bedplate for a two-stroke diesel engine, a cylinder block for a four-stroke engine, and a crankcase for a 60-h.p. engine.—R. A. R.

**Notes on Core Blowing Practice.** W. Fearfield. (Institute of British Foundrymen : Foundry Trade Journal, 1949, vol. 87, Oct. 20, pp. 497–498). This is a short account of the factors to be considered in the successful production of cores on blowing machines.—R. E.

**Stimulating Recruits—The Functions of a Moulder.** E. Hugo. (Neue Giesserei, 1949, vol. 36, Sept., pp. 286–288). An outline is given of the training a moulder should receive during three years' apprenticeship and the kind of examination he should pass on completing it.—R. A. R.

**The Strength of Cores at Elevated Temperatures.** F. Roll. (Neue Giesserei, 1949, vol. 36, Sept., pp. 266–269). The results of compression tests at temperatures up to 300° C. on German core sands using clay, sulphite lye, dextrine, pitch, an emulsion binder, and linseed oil as binders are presented and indicate low and approximately equal strengths with sulphite lye and coal tar pitch, and that the strength increased in the order dextrine, emulsion binder, and linseed oil. The contraction and expansion of cores are also discussed.—R. A. R.

**Portable Electric Driers for Moulds.** A. Bruck. (Metalurgia y Electricidad, 1949, vol. 13, Nov., pp. 59–60). [In Spanish]. Hot gas drying of moulds can be uneconomical. Good drying means total extraction of moisture from the refractory mass. The hot gases must not only pass over the surface of the mould, but must circulate throughout the entire mass of sand. This is the principle of the Brown Boveri portable drier which consists of three parts, the electric air heater, the motor and fan, and the control. The characteristics, power consumption, drying time, and operation of the drier are described briefly.—R. S.

**Manufacture of Ingot Moulds in Cement Sand.** J. G. P. Bruschke. (International Foundry Congress, Amsterdam, 1949 : Foundry Trade Journal, 1949, vol. 87, Dec. 15, pp. 705–708). An illustrated account is given of the operations involved in the moulding of ingot moulds of the slab type. R. E.

**Automatic Moulding.** A. G. Thomson. (Foundry Trade Journal, 1949, vol. 87, Dec. 1, pp. 654–656, 660). Illustrated particulars are given of automatic moulding machines in operation in the foundries of Henry Wallwork and Co., Ltd., Manchester, and Dobson and Barlow, Bolton.—R. E.

**Making a Large Multi-Cylinder Diesel Crankcase.** J. F. Barnes and F. E. Ironmonger. (Institute of British Foundrymen : Foundry Trade Journal, 1949, vol. 87, Nov. 10, pp. 565–570 ; Nov. 17, pp. 593–607). A detailed illustrated account is given of the core-making and moulding practice for the production of a large multi-cylinder diesel crankcase at the works of Ruston and Hornsby Ltd., Lincoln.—R. E.

**The Moulding of a Chemical Pot by a Loam Moulding Method.** J. A. Taylor. (Proceedings of the Institute of Australian Foundrymen, 1944–1947, vol. 4, pp. 277–286). A detailed account is given of the method adopted for preparing the mould for a cast-iron pot 8 ft. in dia. and 6 ft. deep, of hemispherical shape.—R. A. R.

**Moulding a Bedplate for a Twin Briquette Press.** W. A. Stanway. (Proceedings of the Institute of Australian Foundrymen, 1944–1947, vol. 4, pp. 339–345). A description is given of the moulding technique adopted for making two 21-ton iron castings which, joined together, form the frame of a coal-briquetting press carrying a two-throw crankshaft (6½ tons) and a flywheel (19 tons) with connecting rods, crossheads, briquette moulds, etc.—R. A. R.

**Centrifugal Castings.** T. R. Twigger. (British Engineering, 1949, vol. 32, Nov., pp. 728–733). A description is given of the methods developed by the British Piston Ring Co., Ltd. for making centrifugally cast piston rings, cylinder liners, and valve seat inserts. Special cast irons have been developed including ' Britest,' a high-chromium iron with a tensile strength of 28 tons/sq. in. for use where high wall pressures are required, ' Brimol,' a high-alloy austenitic nickel–copper–chromium iron, and ' Brivadium ' which has additions of nickel and chromium in conjunction with a high phosphorus content.—R. A. R.

**Continuous Casting Processes for Production of Sheets and Billets from Ferrous and Non-Ferrous Metals.** J. Chvojka. (Hutnické Listy, 1949, vol. 4, June, pp. 175–182). [In Czech]. Continuous casting methods for ferrous and non-ferrous metals are briefly described, and the history of continuous casting is reviewed. The following methods are mentioned : Junghans, Junghans-Rossi, Williams, Eldred, Poland-Lindner-Betterton, Alcoa-Ennor, Vereinigte Leichtmetallwerke, Jacquet, Grenville-Mellen, Ter-Akopoff, Dow Chemical Co., Reynolds Metals Co., United States Steel Co., International Nickel Co., and Bethlehem Steel Co. The above methods are all based on the casting of the metal in bottomless ingot moulds. A second group consists of those utilizing centrifugal force to obtain the desired shape of the metal. These are : The Bessemer, Hazelett, Merle, Novo-Krematorsk and Ulitovský, and Swiss methods of centrifugal casting.—E. G.

**Machine Tool Castings. Part I—Engineering Requirements.** W. J. C. Paterson. (Proceedings of the Institute of Australian Foundrymen, 1944–1947, vol. 4, pp. 131–146). Co-operation between buyer, designer, and foundryman in the planning of structures requiring castings is strongly urged, and some important requirements in the case of casting (*a*) a planing machine bed 26 ft. long, (*b*) a planer table 17 ft. long, (*c*) straightedges from 2 to 7 ft. long, and (*d*) a hydraulic cylinder and ram 20 in. in dia. × 4 ft. 6 in. high are discussed.—R. A. R.

**Machine Tool Castings. Part II—Method of Manufacture.** W. T. Main. (Proceedings of the Institute of Australian Foundrymen, 1944–1947, vol. 4, pp. 147–162). The moulding, melting, and casting practice for making the bed, table, and uprights for a 26-ft. planing machine are described.—R. A. R.

**Machine Tool Castings. Part III—Metallurgical Aspects.** J. G. Ritchie. (Proceedings of the Australian Institute of Foundrymen, 1944–1947, vol. 4, pp. 163–190). The theory of the structure of cast iron, the changes in hardness with different structures and the effects of section thickness, total carbon, silicon, and phosphorus are explained. On this basis the reasons for the selection of particular irons for making the planing machine castings referred to in Parts I and II are given (*see* preceding abstracts).—R. A. R.

**How to Select Materials for Plastic Mold Cavities.** L. J. Morrison. (Materials and Methods, 1949, vol. 30, Nov., pp. 61–63). The advantages and disadvantages of different alloy steels for moulds for die casting are discussed.—R. A. R.

**New Casting Process Combines Features of Other Forming Methods.** T. C. Du Mond. (Materials and Methods, 1949, vol. 30, Nov., pp. 52–54). The Bacco casting process is referred to. The metal is cast in preheated metal dies on which a constant pressure is exerted during solidification. It is claimed that, in comparison with sand-cast metal parts, the machining is reduced by about 60%, the tensile strength is about 10% higher, dimensions can be held within 0·002 in. in most cases, and more gas and impurities are forced out of the metal. The process has so far been used mainly for aluminium, but 0·81% and 0·43% carbon steels have been successfully cast and an exceedingly smooth finish was obtained.—R. A. R.

**Production Aspects of Precision Casting.** A. Whittaker. (Machinery, 1949, vol. 75, Nov. 17, pp. 709–711). The advantages and limitations of precision casting by the lost-wax process are discussed. The following types of defect and their causes are analysed : Incomplete running, casting shrinkage, and surface marking. The production layout is also discussed.—R. F. F.

## HEAT-TREATMENT AND HEAT-TREATMENT FURNACES

**Some Factors Involved in the Hardening and Tempering of Steel.** M. Cohen. (Sheffield Society of Engineers and Metallurgists and the Sheffield Metallurgical Association, Joint Meeting : Metallurgia, 1949, vol. 40, Oct., pp. 308–312). The author discusses the formation of martensite, retained austenite, martempering, tempering, dimensional stability, tempering of high-speed steel, and the heat-treatment of high-carbon, high-chromium steels. He shows, among other things, that as the tempering temperature is increased the process passes through four stages, namely : (1) Decomposition of tetragonal martensite ; (2) decomposition of retained austenite ; (3) formation of cementite ; and (4) formation of complex carbides. X-ray evidence in support of this is cited. Curves showing percentages of austenite and martensite after various heat-treatments, and changes in length with ageing are also presented.—R. A. R.

**Carbon Steels. New Atmosphere for Decarburization-Free Treatment.** P. F. Hancock. (Iron and Steel, 1949, vol. 22, Nov., pp. 509–512). An account is given of an endothermic generator for the production of non-decarburizing atmospheres ; it has no auxiliary apparatus. The principle of operation is for a mixture of town's gas with a small amount of air to be passed over a heated catalyst bed, the time, temperature and gas/air ratio being adjusted so as to promote the breakdown of hydrocarbons without the simultaneous occurrence of the usual combustion reactions. The output capacity is 500 cu. ft./hr. Operational experience is described and potential applications of this type of atmosphere are considered.—J. C. R.

**Precision Heat Treating Small Steel Parts at IBM.** H. Chase. (Iron Age, 1949, vol. 164, Sept. 15, pp. 83–87). At the plant of the International Business Machines Corp., many thousands of small steel parts are heat-treated daily in both salt-bath and controlled atmosphere furnaces. Operations are largely automatic. Induction heating is used for brazing.—J. P. S.

**Precision Hardening High-Speed Tool Steels.** N. K. Koebel. (Iron Age, 1949, vol. 164, Oct. 6, pp. 86–90; Oct. 13, pp. 80–85). This article describes first the '**L**-type' hardening furnace, so-called because the preheating section is at right-angles to the full heating section and quenching section. A protective gas atmosphere is used; the work, high-speed steel milling cutters, etc., is placed in trays and pushed into the preheating zone, at the end of which the trays are pushed sideways into the full heating zone and through this into a water-jacketed cooling chamber where a fan blows cold protective gas over them. It is also possible to withdraw the trays for oil or water-quenching of the contents. It is claimed that the protective gas quench is intermediate in effect between an oil or lead-bath quench and air cooling. Excellent properties in 18/4/1 and 8% molybdenum steels are claimed, with complete freedom from cracking and decarburization.—J. P. S.

**Improved Metallurgical Properties Obtained by Rapid Quenching of High Manganese Steels at Damascus Steel Casting Co.** (Industrial Heating, 1949, vol. 16. Nov., pp. 1940–1946, 2080). Heat-treating equipment for 12–14% manganese steel castings at the foundry of Damascus Steel Casting Co., New Brighton, Pa., is described. The furnace, 15 ft. 8 in. long × 10 ft. 6 in. wide is heated by natural gas with eight burners for overhead firing and four directed through ducts in the car bottom. On completion of the heating and holding time the car is withdrawn and its platform is tilted by a rack and pinion drive so that the load of castings slides off into the adjacent quenching tank. The theory of the heat-treatment is explained.—R. A. R.

**The Control of Distortion in Case-Hardening Steel.** K. F. Hartnell. (Australian Institute of Metals: Australasian Engineer, 1949, Nov. 7, pp. 83–89). The following aspects of controlling distortion in case-carburizing steels are discussed: (1) Correct drawing-up of specifications; (2) selection of the steel to be used, with special reference to grain-size and carbon content; (3) premachining and intermediate annealing; and (4) heat-treatment methods. Experiments are reported in which cooling curves were obtained for the centres of 4-in. mild-steel cylinders ½ in., 1½ in., and 2½ in. in dia. when quenched in brine, water, oil, and molten salt. The results indicated that salt baths at 200–230° C. are as efficient a quenching medium as oil in preventing the transformation of austenite to ferrite and pearlite, necessary for satisfactory hardening.—R. A. R.

**Some Recent Advances in the Treatment of Steel Parts.** J. Pomey, F. Goutel, R. Coudray, and L. Abel. (Société Française de Métallurgie, Oct. 21, 1948: Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 825–842). After discussing the endurance of steel, Hertzian hardness, notch effect, and cementation and stepped quenching, the authors present a magnetic study of isothermal transformations in the surface layers of carburized or 'carbonitrided' steels; the test apparatus, in which wire specimens were examined, the temperature of quenching and of holding, the choice of the carbon content of the steel in order that the incubation period should be the same as the time required for thermal equilibrium to be reached, and the two regions in the carburized zone, are dealt with in detail. The rôle of nitrogen in carbonitriding followed by quenching, the treatment of residual austenite, and the measurement of superficial stresses are also considered.—A. E. C.

**Production Economies Realized by Proper Use of Induction Heating.** J. A. Evans. (Materials and Methods, 1949, vol. 30, Nov., pp. 57–60). Equipment for the induction hardening of automobile parts is described and illustrated; the examples include cam shafts, the ends of valve lifter rods, brake arms, and internal splines on shafts.—R. A. R.

**Large Annealing Furnace Accommodates Two Railroad Cars at the General American Transportation Corp. Plant.** (Industrial Heating, 1949, vol. 16, Nov., pp. 1950–1956). A very large gas-fired furnace primarily intended for annealing welded tanks for railway tank wagons is described. Its inside dimensions are 52 ft. long, 19 ft. wide, and 21 ft. high from the platform of the car bottom to the centre of the arch. The walls are inclined outwards at 7½° and the weight of the 21-ft. arch is taken by a steel structure, not by the walls. The 12 burners on each enable the furnace and charge to be brought up to 1600° F. in 3 hr.

**The Bright Annealing of Cold Rolled Steel Strip in the Continuous Furnace with Protective Atmosphere.** F. Müller and F. Frutschnigg. (Betrieb und Fertigung, 1949, vol. 3, Nov., pp. 173–178). A description is given of a continuous annealing plant for steel strip which has recently been erected at an Austrian steelworks. A protective atmosphere is prepared by burning, drying, cleaning, and desulphurizing the gas from two small wood-consuming producers. The strip passes through a heating muffle and cooling chamber 500 mm. wide with a total length of 23·2 m. at a speed which can be controlled over a wide range up to 36·45 m./min. The annealing capacity is about 250 kg./hr. of strip with electricity consumption at 220–250 kWh./ton and a wood consumption of 10–12 kg./ton of strip.—R. A. R.

**A Quench Cracking Susceptibility Test for Hollow Cylinders.** C. Wells, C. F. Sawyer, I. Broverman, and R. F. Mehl. (American Society for Metals, Oct., 1949, Preprint No. 13). The inner and outer surfaces of notched specimens from heat-treated hollow steel cylinders for gun tubes were spray-quenched in a jig and the minimum notch depth in inches needed to cause cracking was determined. This minimum is an index of cracking susceptibility. Preliminary data suggest the possibility of using the test as a means of separating tubes which should receive a special quenching treatment to prevent cracking from those which are extremely unlikely to crack in normal commercial quenching practice.

**An Engineering Analysis of the Problem of Quench Cracking in Steel.** J. W. Spretnak and C. Wells. (American Society for Metals, Oct., 1949, Preprint No. 14). This is the second of a series of papers on the quench cracking of hollow steel cylinders for gun tubes (*see* preceding abstract). It presents the results of a field analysis of the problem, carried out to determine (*a*) the nature of the problem as it occurs in actual production, and (*b*) which of the many variables are of particular importance and should be studied. The investigation was carried out by means of statistical analysis of pertinent processing data, starting from the pouring of the ingot up to and including the quenching operation. Three general types of crack were encountered: (*a*) On the bore surface, (*b*) on the outside surface, and (*c*) through the wall of the cylinder The effect of position in the ingot on cracking susceptibility was found to be significant. Cracking occurred mainly in cylinders produced from bottom thirds of ingots. It was confirmed that carbon has a potent effect on susceptibility to cracking. In the analysis of an entire practice, a low degree of correlation was found between the occurrence of cracking and the content of alloying elements (sum of manganese, chromium, and molybdenum contents) in the heat.

**Pre-Bore Quench for Hollow Cylinders.** J. W. Spretnak and C. C. Busby. (American Society for Metals, Oct., 1949, Preprint No. 15). This is the third of a series of papers on the quench-cracking of hollow steel cylinders for gun tubes (*see* the two preceding abstracts). Pre-bore quenching (sometimes called 'bore-lead' quenching) involves a water quench of the bore for a certain period of time, before the simultaneous quenching of both bore and outer surface. As the pre-bore time is increased, the probability of quench-cracking unnotched bore material in a hollow cylinder rapidly decreases, while the probability of cracking outer material first increases, passes through a maximum, and then decreases.

## FORGING, STAMPING, DRAWING, AND PRESSING

**Fundamentals of Drop Forging Design.** (Metal Treatment and Drop Forging, 1949, vol. 16, Winter Issue, pp. 219–224). The importance of die design relative to the rate of production and die life is considered. The influence of the position of the parting plane first in simple basic shapes and then in more complicated shapes is dealt with at length. Finally, die tolerances, machining allowances, and die wear are discussed. R. A. R.

**Press Forgings, New High Production Plant Installed at Bromsgrove.** (Iron and Steel, 1949, vol. 22, Oct., pp. 473–476). An illustrated description is presented of the new B.5 plant of John Garrington and Sons, Ltd., Bromsgrove. The forge bay contains 12 high-speed mechanical presses, one of

which is a No. 7½ Maxipres vertical forging press capable of exerting a load of some 5000 tons.—J. C. R.

**Flame Spinning. A Method of Forming Steel and Other Metals.** K. Harper. (Iron and Steel, 1949, vol. 22, Oct., pp. 448–450). An illustrated description is given of the flame spinning process in which the metal is rotated in a spindle or chuck and, while rotating, it is brought to a forging temperature and formed to the desired contour by a specially designed tool. Reference is made to different methods of rotating the work-piece, factors affecting the speed of rotation, the requirements of the burner, and the types of tool used.—J. C. R.

**How Modern Mechanical Presses Operate.** S. D. Brootzkoos. (Machinist, 1950, vol. 94, Mar. 4, pp. 289–304). The author discusses the construction of presses and the mechanisms and parts that can be found in each type of press.—R. F. F.

**Economies of Short-Run Stamping Practice.** O. T. Andersen and J. M. Andersen. (Iron Age, 1949, vol. 164, Oct. 6, pp. 91–94). The advantages are described of using stamped components, as against components cast or manufactured in other ways, even when only a small quantity is required. A cheap and reliable die manufacturing technique is referred to, but not described.—J. P. S.

**Weir Drawing Machines, Application of " VSG " Hydraulic Variable-Speed Drives.** (Iron and Steel, 1949, vol. 22, Oct., pp. 461–462, 468). *See* Journ. I. and S.I., 1949, vol. 163, Dec., p. 465.

## ROLLING-MILL PRACTICE

**Iron and Billet Mill.** (Iron Age, 1949, vol. 164, Sept. 15, pp. 78–79). The Colorado Fuel and Iron Corp. has just installed a new rod and billet mill at the Minnequa Works, Pueblo, Colo. Billets 4⅛ in. square are rolled down to $\frac{7}{16}$–$\frac{9}{16}$ in., billets 5 in. square are used for larger sizes. The coiling reels can handle coil weights up to 1000 lb.—J. P. S.

**Rolled Bars. Part III—Application of Spread Calculation to Diamond Passes.** A. E. Lendl. (Iron and Steel, 1949, vol. 22, Nov., pp. 499–501). Continuation of a series of articles (*see* Journ. I. and S.I, 1949, vol. 161, Apr., p. 380). In this part the author deals with diamond passes and their intermediate squares and also discusses the diamond–diamond sequence. The amount of the reduction depends upon the angle of the diamond, and if the angle is maintained, the intensity of the reduction decreases with the size of the section.—J. C. R.

**The Determination of Spread in the Hot-Rolling of Steel by Smooth Rolls.** W. Lueg. (Iron and Steel Institute, 1950, Translation Series, No. 394). This is an English translation of a paper which appeared in Archiv für das Eisenhüttenwesen, 1949, vol. 20, Jan.–Feb., pp. 59–68 ; *see* Journ. I. and S.I., 1949, vol. 163, Sept., p. 101.

**Developments in the Last Decade in the Cold Rolling of Steel Strip.** A. Pomp. (Stahl und Eisen, 1949, vol. 69, Nov. 24, pp. 863–879). The developments and improvements which have taken place in Germany and other countries during the last decade in the cold rolling of steel strip are reviewed. Ancillary processes such as pickling, annealing, slitting, galvanizing, and tinning are also included. There are 300 references.—R. A. R.

**Cold Rolling Tests with Longitudinal Tensile Stresses.** W. Lueg and E. Greiner. (Stahl und Eisen, 1949, vol. 69, Nov. 24, pp. 879–884). Using a Rohn-type 12-roll stand with 10-mm. dia. work rolls equipped with means of measuring roll pressure, the braking and winding tensions, and the torque applied to the upper roll, an investigation of the effect of tension was carried out. A 1·12% carbon steel, a 2·92% silicon steel, a 12·5% chromium steel, and 18/8 stainless steel with initial strip section of 15 × 0·25–0·29 mm. were reduced by different amounts in one pass. It was found that, over the whole range of reductions investigated, the application of tension in increasing amounts on the strip leaving the rolls successively decreased the roll pressure. Applying tension on the entry side scarcely affected the roll pressure with small reductions, but with large reductions the roll pressure was decreased by up to almost 50%. With heavy reductions, tension by braking decreased the roll pressure more than equal tension applied by the coilers. The mean deformation resistance and the torque applied to the mill were affected in a corresponding manner. The total power requirements could be appreciably reduced by applying tension.

The operational advantages of tension rolling are pointed out, and formulæ for calculating the forces involved are derived.—R. A. R.

**Steel Strip Extension Gauge.** (Sheet Metal Industries, 1949, vol. 26, Nov., p. 2348). The amount of reduction given to steel strip in the ' temper pass ' is very important as it controls the final deep-drawing properties. An instrument for measuring this small reduction is described. The output of an A.C. generator driven by the rolls is fed to coils displaced relative to each other across the width of the strip entering the mill stand. These coils produce a ' magnetic pattern ' on the strip the form of which is independent of roll speed. Two ' pick-up ' coils in line with each ' printing ' coil receive the magnetic patterns, which, after amplification, are fed to a phase-selective indicating detector. If the speed of the strip entering and leaving the rolls is the same there is no reduction and the output of the ' pick-up ' coils would be in phase provided the distances between each pair of coils are identical ; the indicating instrument is then at zero. If then the distance between the ingoing pair of coils is set at 1% less than that between the outgoing pair and the roll gap is reduced to the point where the coil outputs are again in phase, a 1% reduction in thickness will be obtained.—R. A. R.

## MACHINERY FOR IRON AND STEEL PLANT

**A Large Scrap-Baling Plant.** (Engineer, 1950, vol. 189, Mar. 10, p. 309). An illustrated description is given of a baling machine, designed by the Loewy Engineering Co., Ltd., for sheet-metal scrap. The pressing container, lined with renewable hard steel wearing plates, is 8 ft. 11 in. long, 3 ft. 9 in. wide, and 4 ft. 4 in. deep. It is closed by a heavy sliding cover which runs on wheels and is locked by wedges. Three hydraulic rams, moving forwards, sideways, and upwards respectively, compress the charge into bales 18 in. wide, 15 in. high, and from 32 to 48 in. long at a rate of one every 3 min.—R. A. R.

**The Oil in Hydraulic Machinery.** G. F. Berg. (Die Technik, 1949, vol. 4, Nov., pp. 499–504). The following aspects of fluids for hydraulic machinery are discussed : (1) The choice of oil or water ; (2) the concept of viscosity ; (3) the properties of oil and the effects of pressure, temperature, leakage, and pressure drop ; and (4) selecting an oil. For general purposes and a pressure of 75–100 kg./sq. cm. an oil with a viscosity of 4·8–6° Engler at 50° C. is recommended, whilst for low pressure (10–30 kg./sq. cm. 1·8° to 3° Engler, and for high pressure about 8° Engler at 50° C. are proposed.—R. A. R.

**Vibration-Free Installation of Fixed Machinery.** H. Bürnheim. (Werkstatt und Betrieb, 1949, vol. 82, Oct., pp. 345–348). Recommendations are made on the design of sprung foundations for machinery liable to cause vibration.—R. A. R.

## WELDING AND FLAME-CUTTING

**' Flakes ' When Arc-Welding with Commercial Electrodes.** F. Bischof. (Werkstatt und Betrieb, 1949, vol. 82, Nov., pp. 397–400). After a general discussion of the factors contributing to the formation of ' flakes ' or ' fish eyes ' in fractured welds, the results of tests are reported in which the following factors were studied : (1) The rate of loading in tensile tests ; (2) annealing the electrodes at 650° C. and 900° C. before use ; and (3) peening the weld. High rates of loading markedly decreased the occurrence of flakes, so also did peening the weld, and these factors combined resulted in no flakes at all. The changes in the composition of the electrode coating, the weld metal, and the slag caused by annealing the electrodes are given in tables. The manganese in the weld metal was reduced from 0·47% to 0·08% when the electrodes were annealed for 15 min. at 200° C. The MnO in the slag also fell from 28·5 to 20·7%.—R. A. R.

**Economies in Steel Accomplished by Welding.** H. G. Taylor. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 214–219). Consideration is given to directions in which welding can be used to effect economies in steel consumption.—J. C. R.

**Twenty One Ways to Lower Arc Welding Costs.** R. Wilson. (Iron Age, 1949, vol. 164, Oct. 6, pp. 105–110). To attain economy in production by welding, care should be taken to use readily weldable material, where possible of standard rolled shapes, and to design the joints so that they are most suitable for the thickness of material, adequately strong, and in particular, accessible. Excess of weld metal should be avoided ; intermittent welding may be adequate for strength,

or small continuous welds may be comparable with heavy intermittent ones. Grinding should be avoided by placing the welds in inconspicuous places. Design should take into account the danger of residual stresses, and balance them out if possible. Edge preparation should be simple, sub-assemblies used wherever possible, and jigs and fixtures used to bring the joints into suitable welding position. Electrode size and type should be suited to the material and the requirements of the joint; arc length should be kept as short as possible. This also applies to the stub-ends of electrodes. Finally, the welding generator should be so chosen that it operates at maximum efficiency.—J. P. S.

**Automatic Hard Facing Speeds Production Over Manual Methods.** W. Price and C. V. Faas. (Materials and Methods, 1949, vol. 30, Nov., pp. 64–65). A standard submerged-arc welding machine has been adopted for the automatic hard-facing of cutter bars for earth-removing equipment. These bars are from 6 to 10 in. wide and up to 9 ft. 8 in. long, of 0·50–0·60% carbon steel. The deposited metal is supplied to the welding machine as a coil of uncoated $\frac{3}{16}$-in. wire alloyed with chromium, manganese, silicon, and molybdenum. R. A. R.

**Ship Repairs by Thermit Welding.** R. A. Forster. (Iron Age, 1949, vol. 164, Oct. 20, pp. 71–73). Defective stern frames on two American merchant ships have been repaired by thermit welding; one of these required 1500 lb. of thermit mixture, the other, where two smaller welds were made, a total of 1475 lb.—J. P. S.

**High-Pressure Vessels for the Oil-Refining and Chemical Industries.** H. B. Fergusson. (Institution of Mechanical Engineers: Engineering, 1950, vol. 169, Feb. 10, pp. 149–150). Problems in the manufacture of high-pressure vessels for oil refineries and methods of solving them are discussed with special reference to welding, stress-relieving, and testing. Automatic welding is used for the main longitudinal and circumferential seams and hand welding for the attachment of all fittings. Submerged-arc welding with only two runs is applied to vessels $1\frac{1}{2}$ in. thick, and multi-run automatic welding for thicker plate. X-rays and gamma rays are extensively used for testing.—R. A. R.

**The Welding of Tubes Internally Coated with Asphalt.** J. H. van Swaal. (Lastechniek, 1949, vol. 15, Nov., pp. 301–303). [In Dutch].

**Applications of Ultrasonic Waves.** H. O. Willrich. (Welding, 1950, vol. 18, Feb., pp. 61–66). This paper covers German investigations to determine whether ultrasonic waves could be applied to spot and arc welding to improve the quality of the weld by influencing the crystal structure formed. It concluded that ultrasonic waves can improve welds if applied in sufficient strength and in the right manner. With resistance welding the practical difficulties do not appear to be too great and the development of apparatus for industrial use should be possible. The application to arc welding is more difficult, but an apparatus offering a feasible approach is described.—R. A. R.

**Welding Stainless Steel.** L. F. Spencer. (Iron Age, 1949, vol. 164, Oct. 20, pp. 57–62; Oct. 27, pp. 69–75). The rôle of titanium and niobium in stabilizing austenitic stainless steels intended for welding is fully explained. The author then goes on to describe the nature of resistance welding, including such techniques as pulsation and projection welding: in the second part of the article he deals with arc-welding and gas-welding of these steels.—J. P. S.

**How to Silver Braze and Solder Stainless Steel.** L. F. Spencer. (Iron Age, 1949, vol. 164, Sept. 15, pp. 69–74). A number of useful brazing alloys for stainless steel are described, ranging from 5% silver, 58% copper, and 37% zinc to 80% silver, 16% copper, and 4% zinc; some of these may contain cadmium, manganese, or nickel. It is important that, if brazing takes place at a temperature within the carbide precipitation zone of austenitic stainless steels, the brazing time should be short and the quench rapid; straight chromium steels which have a tendency to air-harden should be brazed at a temperature below the critical. Types of joint are described and the use of jigs is advised for high production rates. The soldering of stainless steels with lead–tin solder containing 75–90% tin is also outlined.—J. P. S.

**New Metal Bonding Process.** K. S. Meakin. (Metal Treatment and Drop Forging, 1949–50, vol. 16, Winter Issue, pp. 225–228, 230). The Redux process of bonding metal surfaces is based on the use of two separate components, a solution incorporating phenol formaldehyde and a polyvinyl resin in powder form. The following operations are involved in the bonding process: (1) Careful cleaning of the surfaces to be joined; (2) application of the liquid to both surfaces; (3) application of the powder over the liquid-coated surfaces; and (4) bonding of the surfaces under pressure at 140–180° C. to cure the resin.—R. A. R.

## CLEANING AND PICKLING

**Spray Pickling.** H. Toner. (British Engineering, 1949, vol. 32, Aug., pp. 238–243). The spray pickling and washing machines and process developed by Curran Brothers, Ltd., are described and illustrated. The pickling machinery consists of a spraying chamber below which is a reservoir containing acid; an acid-resisting pump draws the acid from the bottom and delivers it under pressure to a series of spray tubes; the metal object passes through the jets of acid so formed, whereupon the acid falls back into the reservoir and is recirculated. Copper and copper alloys can be pickled in a machine fabricated of stainless steel, whereas mild-steel articles require a rubber-lined mild-steel construction. One type of machine is cylindrical and its lower half is divided by partitions radiating from the centre; the objects to be pickled are placed on a rotating grid which carries them through the compartments for treatment with acid and washing sprays. R. A. R.

**Degreasing by Trichlorethylene.** (Product Finishing, 1949, vol. 2, Oct., pp. 20–29). The general principles of cleaning with trichlorethylene are outlined, the properties of this solvent are listed and some plants for batch and continuous operation are described and illustrated.—R. A. R.

**The Proper Application of Electrolytic Polishing to Metal Finishing.** G. E. Gardam. (Metal Treatment and Drop Forging, 1949, vol. 16, Autumn Issue, pp. 146–148). The limitations and advantages of electrolytic polishing of metal articles for appearance are pointed out. The process only acts over small lateral distances and will not level out inequalities even a few hundredths of an inch apart. It should not be applied for an extended period on rough articles in the as-manufactured condition.—R. A. R.

**Wear of Metals by Sand Blasting.** K. Wellinger. (Zeitschrift für Metallkunde, 1949, vol. 4, Oct., pp. 361–364). The wear of tubes of different steels used as the nozzles in sand-blasting equipment and the effect of the incident angle of the jet upon the wear of flat plates were investigated. Increasing the hardness of the nozzle decreased the rate of wear. Two kinds of wear phenomena occur on the plate struck by the sand, namely, frictional wear and impact wear. At a small angle of incidence hard plates have a far greater resistance to frictional wear than unhardened metal, but the mild steel has a slightly superior resistance to impact wear.—R. A. R.

**The Waste Pickle Liquor Problem.** W. G. Imhoff. (Wire and Wire Products, 1949, vol. 24, Nov., pp. 1040–1044, 1058, 1059; Dec., pp. 1127–1129, 1154–1159). The history of the development of pickling is reviewed and the difficulties of dealing with large quantities of waste pickle liquor are pointed out together with the shortcomings of existing methods. A list of patents from 1883 to 1948 occupies several pages.—R. A. R.

**Treatment of Waste Waters from the Pickling of Steel.** G. E. Eden and G. A. Truesdale. (Journal of The Iron and Steel Institute, 1950, vol. 164, Mar., pp. 281–284). Experiments of Gehm on the neutralization of dilute solutions of sulphuric acid by application in upward flow to a bed of limestone grit have been extended to show that the limiting concentration of acid that can be satisfactorily neutralized in this way is dependent on the temperature of the solution. The concentration of sulphuric acid (as $H_2SO_4$) which could be continuously neutralized without causing inactivation of the particles of limestone ranged from 9880 p.p.m. at 6° C., to 4360 p.p.m. at 30·4° C., and at any given temperature appeared to correspond approximately to the concentration of sulphate in a solution of calcium sulphate saturated with respect to the metastable calcium sulphate hemihydrate. When the method was applied on a laboratory scale to neutralization of solutions similar in composition to waste washing waters from sulphuric pickling of steel, iron in the form of ferrous sulphate did not interfere with neutralization of free acid, and was not precipitated in the bed. Although iron in

the form of ferric sulphate did not interfere with neutralization of free acid, hydrolysis of residual ferric sulphate caused an increase in the titratable acidity of effluent from the bed ; some precipitation of ferric iron was brought about by the action of the limestone. The process of application at a high rate of upward flow to a bed of small particles of limestone might be of value in reducing the acidity of waste waters from the pickling of steel, and would have the advantage that large volumes of waste water could be treated in a comparatively small plant.

**Modern Pickling Plants.** R. Aldinger. (Glashütte, 1948, vol. 75, pp. 31–32 : British Abstracts, BI, 1949, Apr., col. 296). A plea is made for the improvement of conditions in the pickling room by (1) efficient fume extraction, (2) satisfactory space heating for comfort and the prevention of acid condensation, and (3) good lighting.

**Continuous Pickling. Descaling Steel Strip for Hot-Rolled Coils.** J. H. Mort. (Iron and Steel, 1949, vol. 22, Nov., pp. 479–485). After considering factors affecting scale formation and its extent the author outlines current practice in the continuous pickling of uncoiled hot-rolled strip. Formulæ are derived for computing the throughput of a pickling line and rate of acid consumption. Nomograms and charts are given for equating slab weight, thickness, volume, and surface area ; for equating coil weight, strip thickness, and superficial area ; for equating strip width and thickness, speed of progression, and throughput per hour ; and for determining throughput in square feet per hour from strip width and speed of progression. The application of these formulæ and graphs is explained and worked examples are provided.—J. C. R.

**The Engineering Implications and Economics of Surface Preparation of Mild Steel Prior to Fabrication.** W. A. Johnson. (Institution of Mechanical Engineers, Nov., 1949, Advance Copy). Steel structures which are subjected to repainting during service are considered, particularly those subject to exposed atmospheric conditions. Alternative methods of surface preparation are examined. Mechanical shot-blasting is the cheapest method for structural steel but is only possible prior to fabrication. The choice of prime coats, which must satisfy several requirements, lies between paint, oil, and metallic coatings. It is believed that suitable paint could be developed. No suitable oil is yet available, and sprayed aluminium is the most suitable of the metallic coatings. Fabrication difficulties require further investigation. The cost of painting a structure during a life of 50 years shows that surface preparation before fabrication is economical.

R. A. R.

**Materials Used in Polishing and Buffing.** (Plating, 1949. vol. 36, Oct., pp. 1012–1018 ; Nov., pp. 1127–1130). This paper gives full details of the General Motors standards for cotton buffs and abrasives, and the procedures for polishing.

**Abrasives—Their Manufacture and Use in Germany during the Period 1939–1945.** (British Intelligence Objectives Sub-Committee Overall Report No. 26, 1949, H.M. Stationery Office). This report summarizes the more important and interesting items of information on German abrasives, gained by teams visiting that country, from the various reports already published ; a list of these reports and relevant documents is appended.—R. A. R.

## PROTECTIVE COATINGS

**The Structure of Electrodeposits.** J. J. Dale. (Australian Institute of Metals : Australasian Engineer, 1949, Nov. 7, pp. 43–49). *See* Journ. I. and S.I., 1950, vol. 164, Apr., p. 495.

**Ediphone Standardized Plating Racks.** P. B. Kasakove. (Iron Age, 1949, vol. 164, Sept. 22, pp. 82–83). Simplified racks made mainly of brass wire are used at the Ediphone Division of Thomas A. Edison Inc., West Orange, N.J., for the nickel, copper, cadmium, zinc, tin, and chromium plating of small metal parts.—J. P. S.

**Cleaning and Phosphating Tractor Parts in One Operation.** L. Williams. (Iron Age, 1949, vol. 164, Oct. 27, pp. 66–68). A compound known as ' Detrex 79 ' is used in the phosphate coating of tractor parts of steel, grey iron, and malleable iron. The work passes on a monorail conveyor through a two-zone sprayer ; in the first zone it is sprayed for 2 min. with ' Detrex 79,' and in the second, for 2 min. with water. This process is completed by drying off in air heated by steam : the work is then ready for spray-painting.—J. P. S.

**Electrometric System for Continuous Control of Reduction of Hexavalent Chromium in Plant Wastes.** J. L. Kelch and A. K. Graham. (Plating, 1949, vol. 36, Oct., pp. 1028–1031, 1049). A practical potentiometric method for the continuous control of the reduction of hexavalent chromium to trivalent chromium is described. Ferrous sulphate in concentrations up to 12–16 oz. per U.S. gallon, stabilized with sulphuric acid to a *p*H of 2·0, reduces the hexavalent chromium efficiently, provided enough free sulphuric acid or equivalent is present to insure a final solution *p*H of 2·5 or lower. The potential of a platinum–calomel electrode system at which complete reduction of chromic acid in various concentrations may be assured is between 490 and 500 millivolts.—R. A. R.

**Anti-Fouling Compositions : Accuracy of Leaching Rate Determinations.** H. Barnes. (Journal of the Society of Chemical Industry, 1949, vol. 68, Oct., pp. 305–308). An outline of the leaching-rate method of studying antifouling compositions is given with notes on the sources of errors in such work. By the ' analysis of variance ' technique, the C.V. (coefficient of variation) for copper leaching rates is calculated from the data of a number of series of compositions. C.V. values for a series of cuprous oxide compositions are compared with those obtained from a series of compositions based on phenarsazine chloride. Analyses are given indicating the variability arising from laboratory work, and this is shown to be comparatively small compared with the total variability.

R. A. R.

**New Method for Measuring the Adhesion of Electroplated Deposits.** M. Bonnemay. (Journal des Recherches du Centre National de la Recherche Scientifique, 1948, pp. 156–160 : Chemical Abstracts, 1949, vol. 43, June 25, col. 4584). The central hole of a hollow steel cylinder is filled with a low-melting-point alloy. The metal to be tested is plated across the end of the cylinder. The alloy is removed by heating, leaving a diaphragm of plating across the end of the cylinder. Oil is pumped into the cylinder and the deformation of the diaphragm is measured as a function of the pressure until the plate begins to break away from the cylinder. A second cylinder, of the same inside diameter as the first, is then placed over the deformed plate. With the pressure at which the breaking-away began maintained under the plate, the pressure on the upper cylinder necessary to restore the plate to its original contact is measured and taken as the adhesive force between the plate and its base.

**Electroplated Coatings for Corrosion Protection.** A. G. Sussex. (Australian Institute of Metals : Australasian Engineer, 1949, Oct. 7, pp. 37–42 ; Electroplating, 1949, vol. 3, Nov., pp. 97–99). The author reviews data on the chemical behaviour of some of the commonly plated metals with special reference to their resistance to atmospheric corrosion. There are 32 references.—R. A. R.

**The Influence of Base Metal Surfaces on the Nature and Characteristics of Electrodeposited Metals.** A. T. Steer. (Australian Institute of Metals : Australasian Engineer, 1949, Sept. 7, pp. 38–53). Metallographic techniques, including the study of micrographs at 2000 diameters enlarged five times, have been applied to examining the effect of surface preparation on the adherence and quality of plated coatings. The behaviour of surfaces when subjected to applied stress, the formation, constitution, and properties of flow layers, the production of a polished surface and its behaviour and influence on deposition are among the aspects discussed.—R. A. R.

**Where Do We Go From Here? Part II—Electronics in Electroplating.** J. B. Kushner. (Metal Finishing, 1949, vol. 47, Sept., pp. 48–51, 74). The circuits of electronic control devices for electroplating operations are described and discussed. The purposes of the apparatus described are : (1) To vary the voltage across the tank according to the area of the work exposed so that the current density is maintained constant as racks are put in and taken out ; (2) to keep the metal content of the bath constant ; and (3) to keep the amount of brightener in the plating tank at the optimum value.

R. A. R.

**Electrolytic Coating of Steel Wire.** K. I. Tulenkov. (Stal, 1947, vol. 7, pp. 741–743 [in Russian] : Chemical Abstracts, 1949, vol. 43, June 25, col. 4583). Electrolytic dipping was tried under various conditions of current and solution composition for the purpose of determining the most favourable conditions for dipping and drawing in a continuous lead-coating furnace. A laboratory set-up for continuous dipping comprised an electrolytic bath, rinsing tank, strike tank,

rinsing tank, lime-solution tank, tube furnace, and washing reel. The electrolytic bath contained a 15–20% solution of sulphuric acid at 40–60°. The anode was made of sheet lead or silicon-containing cast iron. Good results were obtained with A.C. at a c.d. of 150–200 amp./sq. dm. at 12 V. It is advisable to provide a wiper to remove slime off the wire leaving the bath. Equally good results were obtained by treating the wire cathodically ; but anodic treatment gave negative results. The strike solution contained 4–5% copper sulphate and 2–3% sulphuric acid at 15–20°. The lime solution contained 200–300 g./l. of CaO and was kept at 100°. In the tube furnace the wire was dried and heated to 120–150°. The wire was evenly coated with copper and CaO, and could be drawn at 234 m./min.

**Tin Coatings in the Wire Industry.** F. A. Lowenheim and H. E. Hirschland. (Wire and Wire Products, 1949, vol. 24, Nov., pp. 1028–1032, 1068, 1069). Some advantages of electroplating tin on wire as an alternative to hot-dipping are pointed out and several possible baths are briefly described. The potassium stannate bath is dealt with more fully as it is probably the best for the high-speed continuous plating of coils. A process not yet employed on an industrial scale is the dipping of steel wire in a bath of molten stannous chloride diluted with an inert salt such as potassium chloride, the bath temperature being in the 660–850° F. range.—R. A. R.

**Potassium Stannate for Tin Plating.** F. A. Lowenheim. (Wire and Wire Products, 1949, vol. 24, Dec., pp. 1117–1120). It is shown that the use of potassium stannate instead of sodium stannate in alkaline plating baths permits much higher plating speeds to be attained while all the advantages of the alkaline stannate solution are retained. Some typical bath formulations are described.—R. A. R.

**The Present Position of Pickling and Hot-Dip Galvanizing.** R. Haarmann. (Stahl und Eisen, 1949, vol. 69, Oct. 13, pp. 734–739). Modern processes and equipment for pickling and hot-dip galvanizing are reviewed with 50 references to the literature.—R. A. R.

**Corrosion Resistant Materials.** V. Evans. (Industrial Finishing, 1949, vol. 2, Oct., pp. 184–192). Materials for lining pickling and plating tanks and coating plating jigs are considered with special reference to Polythene, Perspex, and unplasticized polyvinylchloride. Polythene sheets $\frac{1}{8}$ in. thick can be welded using a stream of hot nitrogen and a Polythene welding rod. This material can be used for almost all acids and most alkalis at temperatures up to 60–70° C. Perspex is joined with a special cement which is polymerized ; this material is unsuitable for chromium plating tanks but is excellent for bright nickel baths. Polyvinylchloride sheets are very strong and tough, and can be welded like polythene ; it is not yet freely available in Great Britain. The best materials so far developed for jigs are Polythene, polyvinylchloride pastes, and certain vinyl co-polymer pastes.—R. A. R.

**The Protection of Steel by the Powder Process of Flame Spraying.** C. A. Robiette. (Industrial Finishing, 1949, vol. 2, Oct., pp. 220–228). The processes of spraying zinc and aluminium with the Schori pistol are described with details of many successful applications to prevent corrosion of steel in constructional work of all kinds.—R. A. R.

**Mechanised Metal Spraying.** F. Loos. (Lastechniek, 1949, vol. 15, Oct., pp. 294–297). [In Dutch]. A description is given of a metal-spraying machine carrying six sprayers in line, with a roller conveyor system for moving joists and other long members through the machine.—R. A. R.

**How to Select Sprayed Metals.** J. E. Wakefield. (Machinist, 1949, vol. 93, Nov. 26, pp. 1114–1116). Sprayed metals are used as a surface treatment on other materials to provide better wear and corrosion resistance. Their main characteristics such as lubrication, hardness, wear resistance, and shrinkage are compared with those of other metals. The types of metal which can be sprayed are discussed ; these include certain steels, copper and bronze, and nickel and some of its alloys. The applications of sprayed metals are briefly outlined.—R. F. F.

**Prefinishing of Steel for Plating or Enamelling Cuts Production Costs.** K. Rose. (Materials and Methods, 1949, vol. 30, Oct., pp. 67–69). As it is much easier to grind and polish flat metal than shaped or recessed metal, processes have been developed by which sheet metal is cleaned, ground, polished, and coated with a protective synthetic resin before being shaped or pressed. The coating is finally peeled or dissolved off.—R. A. R.

**Furnace Atmospheres in Vitreous Enamelling.** S. E. Ryder and G. W. Culshaw. (Institute of Vitreous Enamellers, Oct., 1949 : Foundry Trade Journal, 1949, vol. 87, Nov. 3, pp. 551–555). The authors describe an investigation to determine the effects of flue gas constituents (oxides of sulphur, carbon dioxide, and water vapour) on the scumming of enamels. From the results it appears that the only constituents of flue gases responsible for scumming during fusion are the sulphur oxides, but further work is necessary to confirm this.—R. E.

**Cementiferous Paints. Part II—Zinc Phosphate Cements.** J. E. O. Mayne. (Journal of The Iron and Steel Institute, 1950, vol. 164, Mar., pp. 289–293). The development of cementiferous paints based on the reaction between zinc dust and certain phosphate solutions is described. The paints dry by the formation of a zinc phosphate cement, and sufficient metallic zinc is left to afford cathodic protection to steel. The paints based on di-potassium and tri-potassium phosphate solutions dry at both high and low humidities. Laboratory tests, by immersion in natural sea water, indicate that they are superior to the earlier types of cementiferous paints based on chlorides (*see* Journ. I. and S.I., 1948, vol. 158, Feb., p. 219), when applied to both pickled and rusty surfaces, as the zinc dust has a longer effective life. The paints are cheap and easy to prepare, but although they have an appreciable storage life, it is recommended that they should be used within a few hours of mixing.

**Determination of Adhesion of Enamel to Steel Sheet.** A. Dietzel and E. Wegner. (Berichte der deutschen keramischen Gesellschaft, 1949, vol. 26, Sept.–Oct., pp. 132–137). The best method of determining the adhesion of all types of enamel to steel sheet is found to be that of L. Stuckert. It is based on the measurement of the ' white content ' of the sheet after mechanical de-enamelling. The enamel is broken off the sheet by bending and the sheet is then pressed between hardwood boards. The steel sheet shows a smooth and silvery surface where there has been bad adhesion of the enamel. Since the maximum ' white content ' $W$ of a ' silver sheet ' is about 60% (related to MgO = 100%), the adhesion of the coating is defined as 60–$W$. After testing a large quantity of material, the following classifications of quality were established : 0–8 bad, 8–12 defective, 16–22 good, over 22 very good. Chemical de-enamelling with NaOH gives lower values.—R. S.

## POWDER METALLURGY

**Sheath Working of Metal Powders.** J. R. Long and E. T. Hayes. (United States Bureau of Mines, Feb., 1949, Report of Investigations No. 4464). A process has been developed for consolidating metal powders difficult to treat by conventional methods of powder metallurgy. The method described was successfully developed for titanium powder, but can be applied to many other metals, *e.g.*, cobalt, nickel, and iron. The titanium powder was packed into an iron tube or box the ends of which were sealed by welding ; this was heated to 700° C. and hot rolled down to the desired thickness. In one case a 20-in. length of 5-in. pipe packed with 12 lb. of powder was forged to 2 in. thick and then hot-rolled at 900° C. and produced a sheet of titanium 7 ft. × 7 in. × 0·1 in., which separated easily from the sheath. The properties of the products are given.—R. A. R.

**Survey of Powder Metallurgical Processes. III—Technology of Powder Metallurgy. (3) Compression. (4) Sintering.** L. Auguet y Durán. (Revista de Ciencia Aplicada, 1949, vol. 3, Sept.–Oct., pp. 332–345 ; Nov.–Dec., pp. 413–429). [In Spanish]. The author examines the factors influencing compression and the formation of the compact. These are plasticity of the powder, size and distribution of the powder, surface conditions and porosity of the particles, oxides and other impurities in the powder, addition of extraneous bodies, pressure, rate of compression, period of maximum pressure, and the action of heat. He also considers the principles of sintering and discusses furnaces, protective media, and atmospheres.—R. S.

**The Analysis of Grain Size by Sedimentation with Application to Powder Metallurgy.** T. C. Nguyen. (Annales de Radioelectricité, 1948, vol. 3, Apr., pp. 115–123 : Physics Abstracts, 1949, vol. 52, Aug., p. 509). The methods used are the pipette and the rate of deposition measured by a balance. In the first method the Andreasen pipette has been modified and in the second an automatically registering balance is employed. Experimental precautions are discussed.

**Powder Metallurgy—Principles, Prospects, and Tasks for the Future.** H. Schrader and H. Fahlenbrach. (Zeitschrift des Vereines deutscher Ingenieure, 1949, vol. 91, Oct. 1, pp. 485–492). This review indicates the state of development now attained by various powder-metallurgy processes and describes the properties of hard metals, magnets, and composite materials such as electrical contacts which have been made. Modern applications include making bimetallic strips for temperature controllers.—R. A. R.

## PROPERTIES AND TESTS

**Tensile Fillet Stresses in Loaded Projections.** R. B. Heywood. (Proceedings of the Institution of Mechanical Engineers, 1948, vol. 159, War Emergency Issue No. 45, pp. 384–398). In loaded projections such as the screwthread, gear tooth, and simple cantilever, the strength frequently depends on the fillets connecting the projection to its base, hence the maximum tensile fillet strength may be a critical factor. In this paper an empirical fillet stress formula is advanced which agrees well with experiment over a wide range of projection shapes, and provides a powerful method of determining the strongest shape of projection for any given purpose.—R. A. R.

**A Bar Bend Test and Its Application to Stainless Steel.** C. A. Zapffe, R. L. Phebus, and F. K. Landgraf. (American Society for Metals, Oct. 1949, Preprint No. 24). A description is given of a rod and bar bend-testing machine in which the specimen is inserted in a die and bent at a constant rate of 5°/sec. until it fails (not necessarily fractures). Specimens of 4 in. length of commercial stainless steels in the hot-rolled, cold drawn, and ground conditions were tested to find which steels failed on bending and in what condition. Tests were carried out to study the effect of hardening treatment; and to disclose the nature of the recovery of bendability during stress relieving, tempering, and annealing treatments of the fully hardened steel. The results obtained are discussed and illustrated in the form of graphs.—J. C. R.

**Torsional Stress in Close-Coiled Helical Springs.** A. Morley. (Engineering, 1950, vol. 169, Mar. 3, pp. 231–232). The effect of curvature in modifying the calculation of shear stress in the wire of a helical spring has long been neglected, but formulæ for this have been published by Wahl and by L. E. Adams. The latter derived formulæ for stress and deflection which, subject to implied assumptions, were exact but very cumbersome. The present author sets out to produce a formula equally exact but simpler; he makes clear wherein the calculations are based on assumptions which cannot be entirely justified, and offers an alternative theory.—R. A. R.

**The Frequency of Fracture and the Surface Quality of Helical Springs.** A. Pomp and M. Hempel. (Mitteilungen aus dem Max-Planck-Institut für Eisenforschung No. 505: Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 385–393). The effects of the grinding and coiling processes on the fatigue properties of helical springs have been studied using cylindrical springs 32 mm. in dia. × 62 mm. long, consisting of eight turns of 3·6-mm. wire of oil-hardened 0·67% carbon steel. When failure occurred both 90° and 45° fracture surfaces in one or in two coils of the same spring were found. Most cracks began on the inner side of the coils and most of them occurred in the third, fourth, and fifth coils. The number of reversals to fracture for all degrees of surface quality varied between 0·1 and 10 million. As was expected, rolling and drawing operations had less effect on the frequency of fractures than had defects resulting from grinding and operations affecting the final surface condition.—R. A. R.

**Influence of the Moisture Content of the Air Blast on the Quality of Basic-Bessemer Steel Rails.** J. Godefroy. (Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 869–871). A statistical examination of the results of 30,000 impact tests on basic-Bessemer steel rails, made from 2154 casts, spread over the period from Dec. 31, 1946, to Nov. 19, 1948, showed no connection between the moisture content of the air blast and the number of fissures observed in the impact tests.

A. E. C.

**Impact Stresses in Systems of Bars.** B. N. Cole. (Engineer, 1950, vol. 189, Mar. 3, pp. 264–266; Mar. 10, pp. 288–290). It is still common practice to estimate impact stresses according to the assumption that strain energy is momentarily equal to initial kinetic energy, but this may give an accurate assessment in only very restricted special cases. The medium of operational calculus or that of graphics has been adopted in discussions of impact waves. These methods have disadvantages, and in this paper the author offers a simple non-calculus analysis designed, within its limitations, to compute the maximum stress arising from a single impulsive blow in various systems of bars.—R. A. R.

**An Experimental Investigation of Plastic Stress-Strain Relations.** J. L. M. Morrison and W. M. Shepherd. (Institution of Mechanical Engineers, Dec., 1949, Advance Copy). The paper describes tests carried out on a steel and an aluminium alloy, with the object of discriminating between and assessing the 'incremental' and 'total' plastic strain theories in the region where the elastic and plastic strains are of comparable magnitude. The tests appear to establish that for these two materials the 'total' type of theory is incorrect, and in some cases may lead to large errors. For the types of stress applied, the results strongly support the 'incremental' type of theory, and indicate that a particular variant of this theory (which uses the Mises-Hencky function as the criterion of plastic flow) gives results of satisfactory accuracy.—R. A. R.

**Brittle Fracture in Mild Steel.** J. S. Hoggart. (Australian Institute of Metals: Australasian Engineer, 1949, Nov. 7, pp. 51–62). The engineering and metallurgical factors influencing the susceptibility of mild steel to brittle fracture are considered. After reviewing experimental evidence and theories the author suggests that brittle fracture occurs when the yield strength of the material is greater than the cohesive strength. The ratio of yield strength to cohesive strength is affected by several variables, and is increased by increasing triaxiality of stress, lower temperature, increasing strain rate, cold working, increasing carbon content, decreasing manganese or silicon, strain or quench ageing, and increase in grain-size, elastic superstressing, and preferred orientation. As the limit to which notching may raise the yield stress is two to three times the yield stress in uniaxial tension, a metal is only notch brittle at a particular temperature and strain rate if the ratio of the cohesive strength to the uniaxial yield strength is less than 3 : 1. In conclusion, the economic aspects of decreasing the susceptibility to temper brittleness by control of the composition and grain-size, avoiding preferred orientation, and removing effects of cold work (*e.g.*, planing or oxy-cutting the sheared edge before forming) are dealt with.—R. A. R.

**Some Recent Metallographical and Metallurgical Advances Applied in the Automobile Industry.** J. Pomey. (Régie Nationale des Usines Renault, Apr.–Oct., 1948). The author sets out to show that by the application of the principles of surface hardening, of prestressing, and of accommodation it is possible to get the best service out of the most heavily loaded machine parts. He summarizes modern theories of plastic deformation and of the transformations in steels, and describes modern metallurgical processes that have been based on them and which are employed in the automobile industry.—A. E. C.

**Engineering Steels under Combined Cyclic and Static Stresses.** H. J. Gough. (Institution of Mechanical Engineers, Oct., 1949, Advance Copy). An extensive investigation of the combined effect of a number of variables on the fatigue strength of 12 steels and two cast irons (Silal and Nicrosilal) has been carried out, a complete account of which will be published later; this Presidential Address indicates the field covered and some of the principal conclusions reached. The first stage of the work employed two independent cyclic variables, namely, vertical loads and torsional moments; the second stage employed two further independent variables, static bending stress and static torsional stress. In general, the results are presented as $f/q$ curves where $f$ = range of plane bending stress due to the cyclic flexural moment and $q$ = range of shearing stress due to the cyclic twisting moment. Crankshaft steels covering a wide range of tensile strengths were studied. They included: (1) 0·1% carbon steel; (2) and (3) 0·4% carbon steel (2) normalized and (3) spheroidized; (4) 0·9% carbon pearlitic steel; (5) and (6) 3% and 3–3·5% oil-hardened nickel steels; (7) oil-hardened chromium–vanadium steel; (8) and (9) oil-hardened 3·5% nickel 0·85% chromium steel, one with normal and the other with low impact values; (10) and (11) oil-hardened nickel–chromium–molybdenum steels with 75–80 and 95–105 tons/sq. in. tensile strength respectively; and (12) air-hardened nickel–chromium steel with 95–105 tons/sq. in. tensile strength.—R. A. R.

**The Question of the Uniformity of the Crystalline State in Cold Worked Metals.** E. Brandenburger and F. Staffelbach. (Schweizer Archiv, 1949, vol. 15, Sept., pp. 259–261). X-ray back reflection diagrams have been made before and after cold-deforming steel specimens at different rates. Specimens 10 mm. in dia. were given 10% permanent elongation, in one case in 5¼ min, and in another in 8 sec., the mean crystalline deformation was the same in both cases, but the distribution of the deformation across the section was not uniform, the crystals at the surface being in a much more finely divided state than those at the centre.—R. A. R.

**Large-Scale Torsional Fatigue Testing of Marine Shafting.** S. F. Dorey. (Proceedings of the Institution of Mechanical Engineers, 1948, vol. 159, War Emergency Issue No. 46, pp. 399–415). The paper describes the design and construction of a new type of torsional fatigue testing machine of the resonance type capable of generating a reversed harmonic torque of up to $\pm 3 \times 10^6$ lb.-in. in a stationary test shaft, at a frequency of 2500 vibrations/min. The results of fatigue tests on 9¾-in. dia. mild steel shafts, and on Meehanite cast-iron specimens 6 in. in dia. are given. The scale effect is discussed on the basis of results of tests on ½-in. dia. specimens on a combined-stress fatigue testing machine at the National Physical Laboratory.—R. A. R.

**Crankshaft Damping.** P. Draminsky. (Proceedings of the Institution of Mechanical Engineers, 1948, vol. 159, War Emergency Issue No. 46, pp. 416–432). The author attempts to give a correct physical explanation of natural damping by torsional vibrations, and to obtain approximate formulæ for precalculation of the damping in any given case. Experimental work with a single-cylinder engine driven by external power and excited to torsional vibrations with a spring-loaded cam disc is described. Simple and practical methods for the calculation of damped vibrations in arbitrary elastic systems and of hysteresis and bearing damping in a single-cylinder engine are also given.—R. A. R.

**Fatigue Characteristics of Electroformed Sheets with and without Iron Backing.** H. K. Herschman and C. Thomas. (Journal of Research of the National Bureau of Standards, 1949, vol. 43, Nov., pp. 477–486). The bending fatigue properties were determined for electroformed sheets of iron–nickel, iron–nickel–chromium, and of composite plates of electroformed iron–nickel sheets bonded with solder or a plastic adhesive to open-hearth iron. The fatigue limit of iron–nickel sheet was decreased when a thin deposit of chromium was applied to the nickel face. Heating the iron–nickel–chromium sheets at 260° C. before machining improved their fatigue properties and further beneficial effects resulted from heating after machining. The fatigue limits of plates bonded with the plastic were significantly higher than those joined by soldering.—R. A. R.

**The Effect of Welding Defects on the Tensile Fatigue Strength of Specimens of Steel St 37.** M. Hempel and H. Möller. (Mitteilungen aus dem Max-Planck-Institut für Eisenforschung, No. 504: Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 375–383). Specimens of 0·06% carbon steel were prepared by welding together two pieces of rectangular section, some with longitudinal and others with transverse welds. These, and unwelded specimens of the same dimensions, were subjected to tensile stresses fluctuating from a constant minimum of 2 kg./sq. mm. to various maxima at 500/min. X-ray photographs of the welds were made before the tests. The fatigue strength depended on the type, number, size, and distribution of the inclusions. The strength (*i.e.*, the lower limit of scatter) of welded specimens with no defects was 30 to 40% lower than that of unwelded specimens in the same surface condition. A few separate pores did not lower the strength, and it required a row or a group of defects to reduce the general quality of the weld. With several, but not too many, defects longitudinally welded specimens had a higher fatigue strength than those with similar transverse welds. On the other hand, the strengths of longitudinally and transversely welded specimens were in each case equal when the welds had no defects, a few defects, or a great many defects. It must not be assumed that, when the X-ray photographs of two welds in steel St 37 are similar, the tensile fatigue strengths will be the same, as it is also necessary for the welding techniques, electrodes, and other conditions to be comparable.—R. A. R.

**Testing the Hardness of Metallic Materials.** R. Mitsche and Emma-Maria Onitsch. (Betrieb und Fertigung, 1949, vol. 3, Oct., pp. 157–161). The effect of vibration by hammer blows on Vickers hardness tests on materials ranging from ferrite to martensite has been investigated. The effect is very marked in reducing the reading obtained, especially with the first blow (the table on which the tests were carried out was struck repeatedly with a hand hammer) and this effect decreases with increasing initial load. The practical conclusion is that every precaution must be taken to reduce, or if possible eliminate, vibration during testing, especially with thin specimens.—R. A. R.

**Influence of Composition on Temper Brittleness in Alloy Steels.** A. P. Taber, J. F. Thorlin, and J. F. Wallace. (American Society for Metals, Oct., 1949, Preprint No. 22). An account is presented of an investigation to determine the influence of various amounts of chromium, manganese, nickel, and molybdenum on temper brittleness in low and medium alloy steels. The significant variables, such as grain-size, hardness, microstructure, steelmaking practice, forging direction and reduction, tempering temperature, and method of embrittlement, were maintained as constant as possible. The non-embrittled specimens were water-quenched and the embrittled samples furnace-cooled from the tempering temperature at a rate of 18° C./hr. Qualitative evaluation of the susceptibility of the steels to embrittlement from this type of treatment was determined by a method based on the areas between the energy transition curves from ductile to brittle failure obtained by testing the non-embrittled and embrittled impact bar specimens over a range of temperatures. The effect of individual elements was obtained by comparing impact test results when an element was varied in a series of steels of the same base analysis. Chromium considerably increased the susceptibility of alloy steels to temper brittleness and enhanced embrittlement caused by additions of manganese and nickel. Manganese up to 0·6–0·7% had little effect, but higher contents resulted in temper brittleness as great as that which chromium caused. Nickel and molybdenum (up to 0·25%) decreased susceptibility to temper brittleness. A steel with 0·6–0·7% manganese and at least 0·25% molybdenum produced maximum hardenability with minimum temper brittleness.

**Isothermal Temper Embrittlement.** L. D. Jaffe and D. C. Buffum. (American Society for Metals, Oct., 1949, Preprint No. 23). From data derived from tests on steel SAE 3140, a time–temperature–transformation diagram was determined, the temperature of transition from ductile to brittle failure in the Charpy test being employed as a measure of the extent of transformation. It was found that: (1) Temper brittleness could develop in steels at temperatures up to $Ae_1$. (2) Temper brittleness was not reduced by increasing the time (up to 240 hr.) the steel was held at any temperature below $Ae_1$. (3) When embrittling temperature was plotted against embrittling time, for constant temperature of transition from brittle to ductile fracture, a **C**-shaped curve, which might have a double nose, was obtained. (4) Embrittlement took place during continuous cooling from the tempering temperature to a greater extent than was anticipated from the isothermal data, assuming the effects to be additive.

**The Sliding Wear of Cast Iron.** Seidel and Tauscher. (Die Technik, 1949, vol. 4, Oct., pp. 455–458). With a view to studying the possibility of using cast iron as a bearing metal, a series of wear tests was carried out on 13 grey cast irons. The results indicate the changes in wear resistance with different contents of graphitic and combined carbon, silicon, and phosphorus, and different microstructures. The composition with the maximum wear resistance was: Combined carbon 0·7–0·8%, silicon 1·8–2·2%, and phosphorus 0·4–0·6%, with the total for these three items between 3·0 and 3·3%.—R. A. R.

**The Detection of Fissures by Sweating.** H. de Leiris. (Société Française de Métallurgie, June 23, 1948: Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 797–802). The 'sweating' method of crack detection can be carried out in three ways: (*a*) The piece is immersed in hot oil, the surface is freed from superfluous oil and coated with a layer of finely powdered chalk; the oil in the fissures oozes out and marks the white surface. (*b*) A liquid with fluorescent properties is used; the part is subsequently examined in the dark under conditions such that the exuding fluorescent liquid can be seen. (*c*) In the third method an intensely coloured liquid is used and the surface is dusted with fine talc powder; this process is described in detail.—A. E. C.

**Thermomagnetic Study of the Stability of Cementite.** A. Michel and R. Bernier. (Société Française de Métallurgie, Oct. 21, 1948 : Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 821–823). Thermomagnetic analyses were made on samples of very pure iron carbide which were examined without treatment, or after heating for various periods, to break down the carbide to varying degrees ; the extent of decomposition was indicated by the positions of the magnetization/temperature curves. In a vacuum, as the temperature is raised, the magnetization decreases at the Curie point and disappears at 770° C. ; on cooling, it reappears weakly at 682–703° C., depending on the amount of carbide present, and regains its original value at the Curie point. In a hydrogen atmosphere on heating there is a sharp increase of magnetization at 590° C. followed by its disappearance at 764° C. ; on cooling, the magnetization returns in full strength at 738° C. The reason is that the hydrogen has decarburized the cementite, leaving only iron.—A. E. C.

**Metallurgical Factors Affecting the Magnetic and Mechanical Properties of Iron–Cobalt Alloys.** J. K. Stanley. (American Society for Metals, Oct., 1949, Preprint No. 8). The magnetic properties of iron–cobalt alloys in the range of 25 to 50% of cobalt containing 1% of vanadium or 0·7% of chromium were investigated. Saturation was highest (24,000 gauss) for alloys containing 35 to 50% of cobalt. The 50% cobalt alloys had high permeability (over 2000) at H = 10, and coercive force as low as 0·24 oersted. The high saturation makes the material useful for applications where space or weight is at a premium and cost is a secondary matter. Order–disorder phenomena have considerable effect on the magnetostriction, the electrical resistivity, and the ductility of the material. The iron–cobalt diagram is included in the paper.—R. E.

**Application of Ultrasonic Testing to Steel Plant Metallurgical Control.** J. V. Russell. (Non-Destructive Testing, 1949, vol. 8, Autumn Issue, pp. 7–11). Having explained the principles of ultrasonic testing, the author describes the equipment and process developed by the Republic Steel Corporation for testing billets. Some kinds of defect are always detected by this method ; examples are : Flakes or small flat internal cracks ; pipe, secondary pipe, and shrinkage cavities ; and slag inclusions. When 20-in. square blooms were cooled directly from rolling so as to contain numerous patches of blocky ferrite, there was very poor penetration of the waves, but when there were no large patches of ferrite, moderately good transmission was obtained ; these blooms had a large grain-size. A third bloom, which had recrystallized to a small grain-size, had excellent transmission. Sizes larger than 24 in. presented great difficulty with the apparatus used.—R. A. R.

**Ultrasonic Testing.** D. Erdman. (Non-Destructive Testing, 1949, vol. 8, Autumn Issue, pp. 28–30). Several ultrasonic testing techniques are described, but particular attention is given to pulsation testing with the part and the transmitter immersed in water. Large turbine rotors are inspected in this way, which allows the angle of the sonic beam to be adjusted for maximum echo. When a rubber mask with a ¼-in. hole is moved over the part and scanning takes place through the hole, the position of a defect can be marked on the surface of the metal with a wax pencil. An automatic scanning head to traverse back and forth across a billet is under construction.—R. A. R.

**An Ultrasonic Apparatus for the Non-Destructive Testing of Material.** H. Krautkrämer, J. Krautkrämer, and O. Rüdiger. (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 355–358). An ultrasonic apparatus of high resolving power capable of detecting 0·5-mm. defects in specimens 3 mm. thick is described. The quartz wave transmitter, the specimen, and the quartz receiver are immersed in a water tank. The oscillations received are amplified and and converted to light oscillations which produce a record on sensitive paper.—R. A. R.

**General Material, Process and Quality Control by Non-Destructive Testing.** M. J. Parker. (Non-Destructive Testing, 1949, vol. 8, Autumn Issue, pp. 15–17). The author describes the non-destructive tests used by a company making aircraft parts, and points out when it is advisable to use magnetic testing and when to use X-rays, also at which stages in the several processes of manufacture the tests should be applied. R. A. R.

**Stress Analysis by X-Ray Diffraction.** C. R. Lewis. (Non-Destructive Testing, 1949, vol. 8, Autumn Issue, pp. 18–22). The theory for the application of X-rays to determine residual stresses at the surface of materials is explained, and the particular technique used by the Chrysler Corporation is described in detail.—R. A. R.

**The Fundamental Action of Intensifying Screens in Gamma Radiography.** E. L. Harrington, H. E. Johns, A. P. Wiles, and C. Garrett. (Non-Destructive Testing, 1949, vol. 8, Autumn Issue, pp. 23–27, 30). The nature of the intensifying action of lead screens and Patterson screens has been investigated for gamma radiography. For the lead screens used it was found that nearly 52% of the blackening of the film is due to electrons ejected from the front screen and nearly 48% to electrons ejected from the back screen. The blackening due to absorption of the radiation by the film itself is small. Similar results were obtained with Patterson screens except that about 10% of the effect of the screens is due to low-energy radiation (light) which was stopped by 0·003 g./sq. cm. of aluminium.—R. A. R.

**Industrial Applications of Radiography in the Inspection of Welds.** L. Mullins. (Sheet Metal Industries, 1949, vol. 26, Oct., pp. 2193–2206). The theoretical and practical aspects of industrial inspection by X-rays and gamma rays are reviewed. X-ray apparatus and the radium bomb are first described with details of the setting up of specimens for exposure and the layout of X-ray exposure rooms and dark rooms. The radiography of welds and castings is then dealt with at length, and finally the significance of radiographic evidence is discussed.—R. A. R.

**Inspection by Radon of Large High-Pressure Vessels.** V. E. Pullin. (Engineer, 1950, vol. 189, Mar. 3, pp. 262–263). The high-energy gamma rays emitted by radon enable it to be used economically for examining welds in large pressure vessels. Examples of radiographs taken of a vessel 25 ft. long, 4 ft. 6 in. in inside dia., and 3 in. thick are shown and the technique and safety precautions are described.—R. A. R.

**Properties at Low Temperatures.** E. C. Rollason. (Welder, 1949, vol. 18, Oct.–Dec., pp. 78–79, 90). The mechanisms of ductile and cleavage fractures are briefly explained and the results of impact tests on all-weld-metal specimens and on butt-welded ½-in. plate at temperatures down to — 60° C. are given. An electrode is recommended for producing ferritic welds for service at — 70° C.—R. A. R.

**Considerations Respecting the Results of Tests on Materials at Low Temperatures and Their Presentation, Having Regard to Experience.** H. Biegler and W. Küntscher. (Die Technik, 1949, vol. 4, Nov., pp. 492–498). The results of impact and bend tests on steels at low temperatures, previously reported in the literature with some of the authors' own work, are presented and discussed with special reference to the effects of notches in welded steels. Recommendations on the selection of material for use at low temperature are made.—R. A. R.

**Designing Metal Parts for High Temperature Service.** F. G. Sefing. (Canadian Metals and Metallurgical Industries, 1949, vol. 12, Nov., pp. 14–17, 36). Examples are cited of the high stresses which are created in metals which are subjected to rapid temperature changes in service, and data on the mechanical properties of a pearlitic steel, a 1·15% nickel cast iron, and several non-ferrous alloys are presented.—R. A. R.

**Factors Affecting the Strength of Metals at High Temperatures.** A. G. Metcalfe. (Metal Treatment and Drop Forging, 1949–50, vol. 16, Winter Issue, pp. 235–246). A scheme is proposed for classifying factors affecting the strength of material at high temperatures. By adopting a simplified picture of the mechanism, three groups of methods are obtained. The first includes all those methods which raise the softening temperature, the second contains all the methods by which the resistance to slip may be raised, and the third incorporates all the methods which decrease the chance of failure in a grain boundary. Finally, an attempt is made to assess the relative importance of each of the factors. It seems certain that, as the temperature is raised, the number of factors available for maintaining strength at high temperature will decrease, so that an increasingly large contribution may be required from solid-solution hardening.—R. A. R.

**Testing the Creep Strength of Ferrous Materials and Non-Ferrous Metals.** A. Krisch. (Mitteilungen aus dem Max-Planck-Institut für Eisenforschung No. 506 : Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 395–399). The merits of different methods of creep testing and the presentation of the results are discussed. It is suggested that the German short-time procedure A117/18 should be retained but that the rate of increase in length should be selected to

suit the material and temperature. In long-time tests the object should be to determine the load which would produce an extension of $10^{-4}\%$ in 900 to 1000 hr., or a permanent elongation of 0·2% after 1000 hr., as well as the load producing fracture after 1000 hr.—R. A. R.

**An X-Ray Study of the Scale Formed on Iron Between 400° and 700° C.** O. A. Tesche. (American Society for Metals Oct. 1949, Preprint No. 12). The author has studied the scale formed on iron when oxidized for 15 min. over a temperature range of 400° to 700° C. It was determined that $Fe_3O_4$ is the only oxide formed up to 625° C. At 650° to 700° C. a double-layered scale was found, FeO lying next to the metal with an overlay of $Fe_3O_4$. The thickness of the FeO layer at 650° C. was 37%, at 675° C. 55%, and at 700° C. 80% of the total scale thickness.

**The Attack of Vanadium-Bearing Fuel-Oil Ash on Heat-Resisting Steels.** P. Schläpfer, P. Amgwerd, and H. Preis. (Schweizer Archiv, 1949, vol. 15, Oct., pp. 291–299). The mechanism and extent of the attack of fuel-oil ash containing vanadium on chromium and nickel–chromium heat-resisting steels (two containing molybdenum, and one cobalt) have been investigated. Ash containing vanadium markedly accelerates the scaling of all the steels tested when the temperature is above the melting point of the ash. Raising the temperature strongly increases the attack. No attack occurs in a reducing atmosphere. The vanadium compounds prevent the formation of, or destroy, the protective scale on heat-resistant steels. Layers permeable to oxygen and with little resistance to scaling are formed. The vanadium compounds apparently act as oxygen carriers, with vanadates as an intermediary stage. With molybdenum-bearing steels, the vanadium compounds promote the formation of molybdenum trioxide which sublimates from the scale leaving it in a weakened state; this form of attack is characterized by deep pinholes.—R. A. R.

**Stability of AISI Alloy Steels at Elevated Temperatures.** A. B. Wilder and J. O. Light. (American Society for Metals, Oct., 1949, Preprint No. 17). The authors have studied the stability of 16 typical AISI alloy steels at 480°, 565°, and 650° C. The steels were appreciably oxidized and decarburized after 10,000 hr. exposure at 650° C. in air. The nickel and nickel–molybdenum steels graphitized at 565° C. and graphite was also observed in the AISI 2320 and 4640 steels after exposure at 480° C. The steels containing chromium did not graphitize after 10,000 hr. exposure at either 480° or 565° C. even though an appreciable amount of aluminium was used in the deoxidation practice and in certain instances relatively large amounts of carbon were present. They were also microstructurally more stable than those without chromium. The chromium–vanadium and chromium–molybdenum steels were the most stable. The Charpy test results of the steels investigated at ambient temperature were not adversely affected by exposure at elevated temperatures. The hardness of the steels was, in general, slightly less after exposure.

**The Solubility of Nitrogen in Iron Melts with Additions of Chromium, Manganese, and Nickel.** H. Wentrup and O. Reif. (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 359–362). The manner in which additions of chromium, manganese, and nickel, separately and in combination, affect the solubility of nitrogen in molten iron at 1600° and 1700° C., and on solidification is described. Chromium and manganese increase the solubility and nickel lowers it. Raising the temperature decreases the solubility, and solidification is also accompanied by the evolution of nitrogen. Adding manganese to an iron–chromium melt very markedly increases the nitrogen solubility, whilst nickel lowers it.—R. A. R.

**Methods of Determining Vapor Pressure of Metals.** R. Speiser and H. L. Johnston. (American Society for Metals, Oct., 1949, Preprint No. 11). The theory and practice of determining the vapour pressure of metals by the Langmuir evaporation method and the Knudsen effusion method are described in detail. Vapour pressure equations used to represent experimental data are given, and methods of calculating the thermodynamic functions from vapour pressures, heat capacities, and spectroscopic data are discussed.

**Determination of the Surface Tension of Molten Metals from the Shape of Stationary Drops.** G. Becker, F. Harders, and H. Kornfeld. (Archiv für das Eisenhüttenwesen, 1949, vol. 20, Nov.–Dec., pp. 363–367). A method, developed by G. Quincke, of determining the surface tension of high-melting-point molten metals from the shape of a stationary drop is described. The surface tensions of molten pig iron at 1100–1200° C. in a high vacuum, of electrolytic copper at 1150–1450° C., and of carbonyl iron at 1510–1700° C. in argon have been determined. The method is practicable and the results are comparable with those obtained by the 'maximum bubble pressure' method. The surface tensions of carbonyl iron was about 1370 dyn./cm., the data having a scatter of about ± 5%. No relationship with temperature in the 1510–1700° C. range was observed.—R. A. R.

**Report of the IRSID Committee on Boron Steels.** (Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 859–868; 1950, vol. 47, Jan., pp. 55–87). The Committee on Boron of the Institut de Recherches de la Sidérurgie, set up under the Chairmanship of G. Delbart, was given the task of checking up on all information on the influence of boron in steels published recently in English-speaking countries. Laboratory and full-scale experiments have been made and analytical methods checked, and the results have been compared with those already published. The report of the committee is divided into three parts as follows:

(1) **A Bibliographical Review of Foreign Researches on Very Low-Boron Steels,** by A. Kohn. (This forms the first part of the present publication.)

(2) **An Examination of Methods of Estimating Boron,** by P. Rocquet. (This will be a separate publication by IRSID.)

(3) **A Study of the Influence of Boron on the S-Curve, the Quenching, and the Mechanical Properties of Some Carbon Steels,** by R. Potaszkin. (The general results of this study are given in the second part of the present publication; in an appendix will be found the details of the tests made on an industrial scale.)—A. E. C.

**Effect of Boron and Kind of Boron Addition upon the Properties of Steel.** R. A. Grange, W. B. Seens, W. S. Holt, and T. M. Garvey. (American Society for Metals, Oct., 1949, Preprint No. 5). Using three types of steels (AISI 1046, 1321, and 4150) the authors investigated the effect of boron, and kind of alloy in which it was added, upon the uniformity of the boron distribution, hardenability, austenite grain coarsening, susceptibility to temper brittleness, and tensile and impact properties in each of several heat-treated conditions. For each grade of steel there was a plain heat and three or four heats to which boron was added by different kinds of ferro-alloy. Comparisons were drawn between (*a*) properties at different locations in each of the thirteen heats, (*b*) heats containing boron with a plain heat of corresponding grade, and effect of different kinds of boron addition.

Properties were found to be as uniform in heats containing boron as in each corresponding boron-free heat. Hardenability was considerably increased by adding a minute amount of boron. The grain-coarsening effect of boron was partially overcome by deoxidation with aluminium and by the addition of boron in a complex ferro-alloy containing titanium and zirconium. With a microstructure of tempered martensite, boron had a small adverse effect upon the mechanical properties at low hardness, and a slightly favourable effect at high hardness. In the partially hardened and tempered condition (mixture of bainite and martensite as quenched) no definite effect of boron upon the mechanical properties was observed; in the unhardened condition boron affected toughness adversely, as measured by notched-bar impact tests. Under certain conditions, boron somewhat increased susceptibility to temper embrittlement. In general, the properties of boron steels were not affected by the type of ferro-alloy with which the boron addition was made.—R. E.

**Characteristics and Properties of Cast Low Chromium–Molybdenum Steels.** N. A. Ziegler, W. L. Meinhart, and J. R. Goldsmith. (American Society for Metals, Oct., 1949, Preprint No. 6). The authors have carried out an investigation of five groups of chromium–molybdenum cast steels containing 0·4, 0·7, 1·25, 2·0, and 3·0% of chromium respectively with several molybdenum contents between 0·4 and 0·8% and carbon contents between 0·05 and 0·30% within each group, to determine their thermal characteristics, microstructures, physical properties, and weldability. The work substantiates the already existing opinion that, when the chromium content in this type of steel exceeds about 1%, the carbon content should be restricted to about 0·20% max. and preferably even lower, to ensure a low degree of thermal sluggishness

associated with satisfactory physical properties. When the chromium content is under 1% higher carbon contents can be tolerated.—R. E.

**Science. Application to the Products of the Steel Industry.** W. Barr. (West of Scotland Iron and Steel Institute : Iron and Steel, 1949, vol. 22, Nov. 22, pp. 519–524). A review is presented of recent developments which have led to improved life and performance of steel products. These include the improvement of the notch ductility of mild steel for shipbuilding and structural work, the evolution of weldable high-tensile steels, steels for high temperature service, and stainless-clad steels.—J. C. R.

**Special Steels for Modern Industry.** K. J. B. Wolfe. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 149–156). The main subjects considered in this review of recent developments are steels for gas turbines and general high-temperature work, precision casting, powder metallurgy, permanent magnet steels and alloys, carbonitriding, drills, and drilling techniques.—J. C. R.

**Steel Quality.** W. W. Stevenson. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 157–158, 184). Steel quality is briefly considered from the two aspects of producing good quality steel and the applications for which improved quality steel is essential.—J. C. R.

**Steel Developments for Structural Markets.** T. Stevenson. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 162–168). Recent developments in the use of steel for structural purposes are discussed.—J. C. R.

**Steel and the Consuming Industries.** C. J. Kavanagh and R. A. Hacking. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 169–184). The many uses of steel are reviewed from the point of view of the consuming industries, and the endeavours of the steel producers to meet the requirements of these industries are discussed.—J. C. R.

**The Value to the User of Research in the Iron and Steel Industry.** J. Woolman. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 198–203). The author reviews the development of steels with improved physical and mechanical properties to meet the requirements of the consuming industries.—J. C. R.

**Technical Cooperation between Producer and User. 1. The Viewpoint of the Motor Industry.** R. G. Cooke. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 204–206). The importance to the motor industry of developments in the production of sheet steel is emphasized.

J. C. R.

**Technical Cooperation between Producer and User. 2. Locomotive Engineers and the Iron and Steel Industry.** O. V. Bulleid. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 207–209, 237).

**Technical Cooperation between Producer and User. 3. Steel's Contribution to the Building Industry.** Sir Frederick Minter. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, p. 210).

**Technical Cooperation between Producer and User. 4. Collaboration between the Steel Producing and Tool Using Industries.** E. Mensforth. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, pp. 211–213). The author discusses the properties and manufacture of high-speed steel, the shape and size of ingots, and heat-treatment, in their relation to performance from the point of view of the tool maker.—J. C. R.

**Stainless Steels.** E. W. Colbeck. (Manchester Association of Engineers, Oct., 1949, Advance Copy). This is a comprehensive review of all the stainless steels classified into the following groups : (1) 12–14% chromium steels ; (2*a*) higher chromium stainless irons with 15–18% of chromium ; (2*b*) higher chromium stainless irons modified by up to 3% of nickel ; (3*a*) 23–30% chromium steels ; (3*b*) nitrogen-treated high chromium steels with 23–28% of chromium ; (4*a*) 18/8 stainless steels ; and (4*b*) modified stainless steels. The equilibrium diagrams and microstructures are shown and their properties and applications discussed.—R. A. R.

**Rotor Forgings for Turbo-Generators.** J. Novák. (Bulletin des schweizer Elektrotechnischen Vereines, 1940, vol. 40, No. 24, pp. 943–951 : [Abridged Translation] Engineers' Digest, 1950, 11, Mar., pp. 72–74). The author discusses the mechanical properties required of rotor forgings, the influence of the slab size on the quality of the forgings, the influence of the microstructure on the mechanical properties and the faults found in the rotor material. He considers that cavities and silicate inclusions are relatively harmless, segregation cracks and flakes can be detected by radial bores and forging cracks in the trunnion by ultrasonic test methods. Transverse cracks can be avoided with modern manufacturing methods and detected by ultrasonic methods. He therefore suggests that axial boring is superfluous as a method of control, and with the removal of this test the high-alloy steels, which are prone to the above fault, can be replaced by the more reliable pure carbon or low-alloy steels and by steels which have better magnetic and welding properties.—R. F. F.

**The Lateral Instability of Yielded Mild Steel Beams of Rectangular Cross-Section.** B. G. Neal. (Philosophical Transactions of the Royal Society of London, Series A, 1950, vol. 242, Jan. 24, pp. 197–252). The phenomenon of lateral buckling in deep mild-steel beams of rectangular cross-section is studied from both a theoretical and an experimental standpoint.—R. A. R.

**Anisotropy, Mechanical Behaviour and Normalization of Grey Cast Irons. Properties of Static Strength and of Rigidity.** A. Collaud. (Von Roll Mitteilungen, 1949, vol. 8, Jan.–Dec., No. 1–4, pp. 3–164). The author is concerned with the problem of the correct normalization of grey cast irons from the point of view of rigidity and static strength. The paper is divided into eight chapters. In the first, he states the problem and discusses the mechanical behaviour, the anisotropy, and the normalization of grey irons. In the second chapter he deals with the anisotropy of graphite and the elastic modulus (Hooke's law and grey iron ; the elastic modulus and the transverse testing of cast irons ; primary anisotropy and the elastic modulus of grey irons). Chapter III deals with the anisotropy of the matrix and the Brinell hardness (the structure of the matrix and the hardness ; the anisotropy of the secondary structure and the hardness ; the relation between the primary and secondary structure of grey irons). In Chapters IV to VII the author deals with the anisotropy of grey irons in relation to the tensile strength, to the transverse strength of long and of short specimens and to the conventional elastic limit ; in each case he reviews the characteristics of the test in question and considers the influence of the diameter of the test-pieces the physico-chemical composition, and the structure on the anisotropy of the property in question. In the last chapter he deals with the anisotropy of the static mechanical behaviour and the normalization of grey irons, and sets out the principles of anisotropy and of adaptation to working conditions. The very large number of experimental results and microstructural observations on which the paper is based are summarized in over 40 tables, 141 diagrams, and 60 micrographs.—A. E. C.

**An Apparatus for Preparing Small Samples of Pure Iron to which Fixed Quantities of Impurities Can Be Added.** J. D. Fast. (Philips Research Reports, 1949, vol. 4, Oct., pp. 370–374). A high-frequency melting apparatus is described for the preparation of alloys of well-defined composition in quantities up to 2 kg.—R. A. R.

## METALLOGRAPHY

**The Critical Use of the Metallurgical Microscope.** L. E. Samuels. (Australian Institute of Metals : Australasian Engineer, 1949, Nov. 7, pp. 134–141). The author discusses those features of the microscope which need to be understood if the microscope is to be operated under optimum conditions and high-quality micrographs obtained, in particular, resolution and sharpness of focus.—R. A. R.

**The Electron Microscope.** A. E. Williams. (Mechanical World, 1949, vol. 126, Oct. 21, pp. 470–474). Descriptions are given of the Plessey, the Metrovik EM3, and the Philips electron microscopes.—R. A. R.

**Metallurgical Achievements of the Electron Microscope.** G. A. Geach. (Metallurgia, 1949, vol. 40, Oct., pp. 319–326). The author reviews the literature from 1933 to 1948 inclusive, on electron microscope techniques of interest to the metallurgist. There are 110 references.—R. A. R.

**Symposium on Metallography in Color (1948).** (American Society for Testing Materials, 1949, June, Special Technical Publication No. 86). This symposium, presented at the June, 1948, Annual Meeting of the A.S.T.M. was sponsored by Subcommittee IV on Photography and Committee E-4 on Metallography. The following papers were presented :

**Metallography in Color.** R. P. Loveland. (Pp. 2–22). This paper, which appeared in 1944 (*see* Journ. I. and S.I., 1944, No. II, p. 135A) is reprinted because of its bearing on the subject.

**Microscope Optics for Color Metallography.** J. R. Benford. (Pp. 23–29). With the increase in interest in colour metallography, the optical designer is led to re-evaluate the optical combinations of the metallurgical microscope. The experimental means used to evaluate optical designs, and the influence of colour work on the design programme are described and discussed.

**Quality and Quantity of Illumination in Color Metallography.** R. P. Loveland. (Pp. 30–50). Although colour pictures must be quite accurately exposed, determination of the quantity of the illumination is relatively simple. The highest illumination in the plane is the best criterion for exposure and the easiest to determine. The reason for this criterion is explained and exposure constants given. Visual photometers can be satisfactorily used and have great sensitivity and range in spite of their relative cheapness. Photoelectric photometers have some advantage. The photographic method is ideal except for the time consumed. Directions for making suitable instruments of both types are given.

**Color Metallography Simplified.** M. Clogg, jun. (Pp. 51–53). A simplified method of making colour micrographs is explained and described. The procedure for balancing the quality of light with liquid filters was tedious and has been greatly simplified with the aid of a colour meter and a special set of glass filters. With these, a 35-mm. film and an adaptor for the camera, the production of colour micrographs becomes relatively inexpensive.

**Some Applications of Color Metallography.** W. D. Forgeng. (Pp. 54–58). Methods for obtaining colour transparencies and colour prints of metallographic specimens are briefly discussed. Details are given of the differentiation of the carbide phases in tool metals and of the recognition of the sigma phase in iron–chromium base alloys by means of etching techniques that have been developed permitting the identification of the metallic constituents by characteristic staining. The use of reflected polarized light and crossed nicol prisms in combination with colour micrography for distinguishing various intermetallic compounds and for delineating the grain-size of alloys is discussed.

**The Application of Color Photography to the Study of Nonmetallic Inclusions.** A. M. Hall and E. E. Fletcher. (Pp. 59–64). The colour phenomena which may be associated with nonmetallic inclusions in metals and alloys are described and their importance is discussed. The manipulation of the microscope for reproducing the colour of the inclusions is explained. It has been found that carbon-arc illumination through an ultraviolet filter lightly tinted with blue, employed in conjunction with apochromatic optics gives satisfactory results. Several illustrations of inclusions are reproduced in colour.—R. A. R.

**Some Peculiarities in the Structure of Grey Cast Iron.** I. Iitaka. (Bulletin of the Institute of Physical and Chemical Research, vol. 21, No. 3, pp. 223–230). [In Japanese].

**Interferometric Study of Metal Surfaces.** S. Tolansky. (Metal Treatment and Drop Forging, 1949–50, vol. 16, Winter Issue, pp. 195–203). An account is given of recently developed optical techniques for studying the topographies of surfaces. These methods can reveal surface contours with height alterations down to the order of molecular dimensions; the cost of the necessary equipment is very small. The basis of the multiple-beam method lies in using the light-interference effect to such advantage that displacements of 0·001 part of a light wave can be measured. The 'crossed' fringe technique for transparencies, and a method using 'fringes of equal chromatic order' for metals are described. A white light source is used in the latter and, instead of viewing the plane of localization, an image of the latter is thrown on the slit of a spectrograph; each fringe is a repetition of its neighbour and each is a profile of a line section of the surface. Many examples of the application of these techniques are reproduced. Fringes across slip bands on a single crystallite of aluminium are shown; these clearly reveal that the adjacent parallel regions are stepped by small amounts, varying from slip to slip, and often varying along their lengths by a small fraction of a light wave. Surface distortions accompanying indentations made in hardness testing have been examined. Opposite each face of a square impression in steel, the metal is seen to have piled up to a height of about 5 or 6 light waves; at the corners the distortion is much less, due to the cutting action of the diamond edges. R. A. R.

**Metallic Segregation in Ferritic Steels.** K. J. B. Wolfe and I. R. Robinson. (Metal Treatment and Drop Forging, 1949, vol. 16, Winter Issue, pp. 209–218). A technique for microradiography and some results obtained are described. The specimens used were normally $0{\cdot}5 \times 0{\cdot}5 \times 0{\cdot}002$ in. They were placed in the camera in contact with a small square of very fine grain photographic plate and a narrow beam of X-rays allowed to pass through the specimen and photographic plate. After processing, the 0·5 in. dia. blackened image was enlarged between 100 and 300 times in a Vickers projection microscope; this image could be photographed to obtain a permanent record. The presence of segregations of manganese, chromium, and tungsten in various ferritic steels was demonstrated. The most serious segregation is that of manganese; this is always sharply defined and dense, whilst segregates associated with chromium, vanadium, and tungsten consist of broad diffuse bands. Molybdenum appears to suppress the segregation of manganese to a noticeable extent.—R. A. R.

**Non-Metallic Inclusions in Steel.** W. Zednik. (Hutnik, 1949, vol. 16, Mar.–Apr., pp. 131–144). [In Polish]. The author gives a short review of non-metallic inclusions and impurities in steels. Most of the examples show non-metallic inclusions in steel deoxidized by the addition of a calcium–silicon alloy and aluminium. None of the methods (chemical, metallographical, or X-ray) can give, alone, all the information about an inclusion. The spectrographic method has not been applied by the author.—W. J. W.

**The Thermodynamics of the Iron–Nickel Alloys.** O. Kubaschewski and O. von Goldbeck. (Transactions of the Faraday Society, 1949, vol. 45, Oct., pp. 948–960). The partial and integral thermodynamic data of the formation of $\gamma$ iron–nickel alloys have been derived from equilibrium measurements between water, hydrogen, and alloys of various composition. The entropy of formation is almost ideal, and the heat of formation is slightly negative. The corresponding data for the alpha phase have been derived from published work on heat-content measurements. Thus the free-energy curves for $\alpha$ and $\gamma$ iron–nickel alloys have been calculated and the accuracy estimated. From these curves the phase boundaries $\alpha/\alpha + \gamma/\gamma$ have been derived. Comparison of the results with the most reliable data from the literature shows good agreement. The results are discussed from a general aspect.

**Secondary Recrystallization of Face-Centred Ni–Fe Alloys.** G. W. Rathenau and J. F. H. Custers. (Philips Research Reports, 1949, vol. 4, Aug., pp. 241–260). The primary texture of severely reduced (by rolling) nickel–iron alloys is the cubic orientation. At high temperatures abnormal grain growth, *i.e.*, secondary recrystallization, may occur; the orientations of the secondaries are different from the cubic and several new orientations have been found. Normal secondary recrystallization is thought to be the grain growth of primary crystals with a high temperature of primary recrystallization. Experimental evidence in support of this is cited.—R. A. R.

**An X-Ray Investigation on the Ternary Equilibra in the Iron–Nickel–Aluminium System.** S. Kiuti. (Science Reports of the Tôhoku Imperial University, 1941, vol. 29, No. 5, pp. 742–794). The author discusses the results of X-ray analysis of the iron–nickel–aluminium permanent-magnet alloys. Although an unstable $\gamma$-phase appears in the early stages of annealing, this disappears with prolonged annealing and X-ray diagrams (by the Seemann-Bohlin method) show the coexistence of the $\alpha$-phase and a new phase ($\alpha'$). The latter was identified as a solid solution of the intermetallic compound NiAl. The author regards the reaction $\alpha \rightarrow \alpha + \alpha'$, not $\alpha \rightarrow \alpha + \gamma$, as the main one.—R. A. R.

**Iron Carbide.** W. Sauerland. (Neue Giesserei, 1949, Sept., Supplement No. 1, pp. 43–44). Entropy calculations are made which show that the energy of the reaction $3Fe + C \rightleftharpoons Fe_3C$ remains positive, at least up to 900° K. (627° C.) which means that it does not take place spontaneously up to this temperature. The motive force of the reaction increases with increasing temperature whilst the equilibrium in this temperature range remains on the 3Fe + C side.—R. A. R.

**Measurement of Retained Austenite in Carbon Steels.** B. L. Averbach, L. S. Castleman, and M. Cohen. (American Society for Metals, Oct., 1949, Preprint No. 20). A preliminary report is presented of an investigation to determine by an X-ray method the retained austenite in low and medium carbon steels. Steels with carbon contents varying from 0·20 to 1·07% were quenched in brine at 20° C. from their austenitizing temperatures, and some specimens were then cooled immediately to − 196° C. in liquid nitrogen. Measurable quantities of austenite were found even below 0·5% carbon, and in such steels comparatively little austenite transformed on subsequent freezing, although with higher carbon contents some of the austenite was converted on freezing.—J. C. R.

**The Effect of Alloying Elements on The Transformation Characteristics of Induction-Heated Steels.** J. F. Libsch, W. P. Chuang, and W. J. Murphy. (American Society for Metals, Oct., 1949, Preprint No. 21). The influence of alloying elements, classified as carbide-forming and ferrite strengthening, on the transformation characteristics of the austenite formed in induction heating is discussed. In plain carbon steels and alloy steels containing ferrite-strengthening elements only, the austenitizing cycle is of minor importance, and the transformation characteristics and properties for hardened and tempered steel are similar to those obtained by conventional furnace heating. The austenitizing cycle is of primary importance in alloy steels containing carbide-forming elements, and austenitizing temperatures 150° to 300° F. in excess of conventional hardening temperatures are required to take full advantage of these elements. Such temperatures may be used without undue grain-coarsening though they will develop deeper cases in surface hardening. It is believed that high abrasion resistance and an optimum balance of strength and toughness may be produced in medium carbon steels by using an induction heating cycle wherein alloy carbides are only partially dissolved.

**Equilibrium Data for Tin Alloys.** (Tin Research Institute, Greenford, 1949). This booklet presents the existing data on the thermal equilibria of the more important tin-containing binary metallic systems in diagram form, 36 diagrams being given. A large number of classified references on all the ternary alloys of tin which so far have been partly or fully investigated are also included.—R. A. R.

## CORROSION

**The Costs of Corrosion and of Its Control.** W. H. J. Vernon. (United Nations Scientific Conference on the Conservation and Utilisation of Resources: Engineering, 1950, vol. 169, Feb. 10, pp. 160–161; Canadian Metals and Metallurgical Industries, 1949, vol. 12, Nov., pp. 24, 25, 34–36). Estimates of the annual cost of iron and steel wasted by rusting are discussed. In the long view, Sir Robert Hadfield's estimate in 1922 of £600,000,000 must be greatly exceeded today. U. R. Evans' formula for the economics of painting is explained, and the corrosion of ships and buried pipelines is dealt with.

R. A. R.

**The Mechanism of Corrosion of Copper Steels.** N. D. Tomashov, G. P. Sinel'shchikova, and M. A. Vedeneeva. (Journal of Physical Chemistry, U.S.S.R., 1949, vol. 23, pp. 289–303: Chemical Abstracts, 1949, vol. 43, Aug. 10, cols. 5722–5723).

**A Comparison of Some Metals for Use in Acid Pickling Baskets.** E. E. Halls. (Sheet Metal Industries, 1949, vol. 26, Oct., pp. 2127–2130, 2136). Weight-loss tests were made with sheet metal specimens of Monel metal, stainless steel, mild steel, aluminium, duralumin, copper, and brass, immersed for various times in typical acid pickling solutions, in order to obtain data on materials for making pickling baskets. In 10% (by volume) $H_2SO_4$ mild steel was very poor, stainless steel rather poor, and Monel metal the best. With 3% (by weight) potassium dichromate in 5% (by volume) $H_2SO_4$ the attack on 18/8 stainless steel was negligible and that on Monel metal, copper, and brass markedly accelerated. In 50% (by volume) HCl, stainless steel and mild steel were poor, and Monel metal outstandingly good. In a 'bright dip' containing $H_2SO_4$, $HNO_3$, a small amount of NaCl, and heavily diluted with water, stainless steel was almost unaffected whilst Monel metal and mild steel were heavily attacked. Stainless steel was resistant to all the nitro-sulphuric acid mixtures tried and to nitric acid itself.—R. A. R.

**Corrosion Tests in the Processing of Soap and Fatty Acids.** W. Z. Friend and J. F. Mason, jun. (Corrosion, 1949, vol. 5, Nov., pp. 355–368). The results are presented of a large number of corrosion tests carried out by several producers of soap and fatty acids. The specimens were discs of many non-ferrous alloys, 11 stainless steels, a 5% chromium iron, a mild steel, and a cast iron. These discs, held in a special fixture, were placed in various units of the works such as soap kettles, caustic soda tanks, brine tanks, in soap lye, salt-glycerine slurry, salt-glycerine evaporators and glycerine treatment tanks, and plant for the treatment of fatty acids.

R. A. R.

**Experience and Economic Benefits from Cathodic Protection on Gas Distribution Systems.** A. W. Peabody and C. L. Woody. (Corrosion, 1949, vol. 5, Nov., pp. 369–377). Descriptions are given of the cathodic protection systems for buried pipelines employed by two gas distribution companies in the southern States of the U.S.A. The costs of five systems are given and it is shown that considerable economic advantages were gained by the installations.—R. A. R.

**Electrolytic Corrosion of Steel in Concrete.** G. M. Magee. (Corrosion, 1949, vol. 5, Nov., pp. 378–382). Field tests are described in which 1-in. steel bars 6 ft. long embedded in concrete of various thicknesses, with and without asphalt protection, were buried leaving 6 in. of steel projecting above ground. These were connected to a 25-V. D.C. supply on the positive side, the negative side being connected to steel rails buried at the corners of the test area. After being continuously charged for 15 months, severe cracking and deterioration of the concrete and corrosion of the bar occurred in all cases with no asphalt protection. Additional tests led to the following conclusions: (1) A.C. potential does not corrode the steel or deteriorate the concrete; (2) the corrosion and deterioration occur when the steel is in plain concrete buried in soil containing sulphate ions, and sufficient direct current flows; (3) these effects can be prevented by completely surrounding the concrete with a waterproof asphalt membrane, or by coating the steel with asphalt to insulate it from the concrete; (4) the mere flow of current through concrete does not deteriorate it; (5) admixtures to the concrete such as emulsified asphalt do not protect it; (6) a sulphate-resisting concrete does not deteriorate, but if the steel corrodes inside it the concrete may crack; and (7) stainless steel bars resist corrosion and help to prevent cracking of the concrete, but if sulphate ions are present in the soil the concrete will deteriorate.—R. A. R.

**Corrosion in Petroleum Processes Employing Aluminium Chloride.** R. S. Treseder and A. Wachter. (Corrosion, 1949, vol. 5, Nov., pp. 383–391). An aluminium-chloride/hydrocarbon complex is employed as a catalyst in commercial processes for making butane and it is to be similar to aqueous hydrochloric acid in its corrosive action on metals. The use of 0·4% antimony trichloride as an inhibitor is discussed and mechanism for the corrosion reaction is proposed.

R. A. R.

**Co-operative Corrosion Testing in the Wood Pulping Industry.** T. R. Gaulke and M. A. Scheil. (Corrosion, 1949, vol. 5, Nov., pp. 392–402). The organization of co-operative corrosion testing in the wood pulp industry is discussed and recommendations are made on the standardization of specimens and methods of exposure. The results of many tests on non-ferrous alloys and stainless steels in sulphate and sulphite solutions and in nitric acid are presented.—R. A. R.

**Stress Corrosion in Radial Flow Steam Turbines.** G. H. Wray. (Engineering, 1950, vol. 169, Feb. 10, pp. 141–143; Feb. 17, pp. 169–171; Feb. 24, pp. 217–218). The paper records investigations on the cold-work effects of rolled alloy steel turbine blade rings which fix the blades in the Brush Ljungstrom double-rotation turbine. This design, which was susceptible to stress corrosion, has been superseded. Specimens ($5 \times \frac{1}{2} \times \frac{1}{4}$ in.) of nickel–chromium–molybdenum steel were clamped in a fixture to produce tension on one side and compression on the other; other specimens were bent and then released so as to produce residual tensile and compressive stresses. These two types were immersed for several days in caustic soda solution at 210° F. In all cases fine cracks of a branching character were found extending into the steel from the side in tension. The results suggested that the stresses producing stress corrosion are essentially tensional in character, and that uneven stress distribution is an

accelerating factor in cracking. Tests with chromium–molybdenum steel also produced these cracks, but in general their extent was less than with nickel–chromium–molybdenum steel. Caustic soda is regarded as the fundamental corroding agent and silica as an agent which tends to accelerate failure by its action as a catalyst. X-ray investigations showed that there was a residual compressive stress of 30 tons/sq. in. at the surface of the ring where it was rolled over to grip the ends of the turbine blades. A satisfactory stress-relieving treatment at 510° C. was arrived at after trials.—R. A. R.

**The Mechanism of the Action of Chromates in the Corrosion of Plain and Alloy Steels in Liquid Media.** L. Cavallaro, A. Indelli, and A. Tani. (Société Française de Métallurgie, Oct. 21, 1948 : Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 818–820). Electrodes of similar metals (mild steel, 12% chromium, 18% chromium, 18/8 chromium–nickel, and 24% chromium steels) were immersed in solutions of $N/10$ sodium sulphate used alone or with additions of sulphuric acid up to $2N$ and 0·1% of potassium chromate, and the influence of the chromate in inhibiting or stimulating cathodic or anodic corrosion was studied. The results are grouped in a table and discussed. It is concluded that no theory of the action of chromates accounts for the moderate anodic inhibition observed even with very low $p$H values with rustless steels, and that relatively low-resistance materials can be used in highly aggressive media if they are protected externally by chromates.—A. E. C.

**Tests on the Resistance of Heat-Resisting Alloys to Corrosion by Waste Gases.** P. Galmiche. (Société Francaise de Métallurgie, Oct. 21, 1948 : Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 843–848). The materials examined were two nickel–chromium and one nickel–molybdenum alloys and five heat-resisting steels of various compositions. They were heated to 800°, 900°, and 1000° C. in a muffle furnace through which were passed air, waste gases of combustion of known composition, and the same waste gases with 1% of $SO_2$ added. The specimens were freed from oxide by electrolytic pickling by Héron's method before determining the loss of weight due to corrosion. In some tests the specimens were cooled down and reheated repeatedly. No table of results is presented, but the behaviour of certain individual specimens and of the steels and nickel alloys as groups is mentioned and discussed. The penetration of corrosion is discussed and illustrated in half-a-dozen micrographs.—A. E. C.

## ANALYSIS

**Determination of Silicon in Cast Iron and Steel by a Volumetric Method.** P. P. Budnikov and S. S. Zhukovskaya. (Journal of Applied Chemistry, U.S.S.R., 1948, vol. 21, pp. 959–961 : Chemical Abstracts, 1949, vol. 43, Aug. 10, col. 5700). The method described is based on the precipitation of $K_2SiF_6$, followed by titration with NaOH.

**Photocolorimetric Determination of Silicon in Pig Iron and Steels.** Iu. I. Usatenko and Iu. Ya. Orlova. (Zavodskaya Laboratoriya, 1949, vol. 15, Nov., pp. 1365–1366). [In Russian]. The photocolorimetric determination of silicon in steel and pig iron is described, in which the reduction of the silico-molybdenum complex is carried out with a 5% aqueous solution of Mohr's salt. The weights of samples of pig iron and steel used for each determination were 0·1 and 0·25 g., respectively, the corresponding ranges of silicon content for which the working curves are given being 3% and 0·75%. Some results of numerous trial determinations with this method are tabulated and indicate its accuracy.—S. K.

**On the Determination of Silicon in Steels by a Photoelectric Method.** T. P. Temirenko. (Zavodskaya Laboratoriya, 1949, vol. 15, Nov., p. 1367). [In Russian]. For the determination of silicon in steel by the method described, 0·1 g. of the sample is dissolved in $3N$ nitric acid and the solution is diluted to 100 ml. To a 5-ml. portion of the liquid, 17 ml. of 0·15 $N$ sulphuric acid and 5 ml. of a solution of ammonium molybdate are added, and the solution is allowed to stand for 3 min. after stirring. 15 ml. of $8N$ sulphuric acid are added, followed, after 1 min., by 4 ml. of 0·5% stannous chloride, and the volume of the solution is made up to 50 ml. with thorough stirring. The colour intensity of the solution is determined photocolorimetrically 6 min. after the addition of the stannous chloride, a red or greenish-yellow filter being used. A blank determination is carried through simultaneously using a silicon-free iron solution.—S. K.

**The Application of Thio-Urea in the Colorimetric Determination of Molybdenum.** L. B. Zaichikova. (Zavodskaya Laboratoriya, 1949, vol. 15, Sept., pp. 1025–1027). [In Russian]. A method for the colorimetric determination of molybdenum in ores is described ; the thiocyanate complex is used and the reduction of molybdenum to the pentavalent state is carried out with thio-urea in the presence of copper as a catalyst. Unlike copper, iron affects the colour of the solution and must be allowed for in the preparation of the colorimetric standards. Tests carried out with standard samples gave results in good agreement with those obtained using stannous chloride as the reducing agent.—S. K.

**On the Detection of Alloying Elements in Steel by the Spot Method.** M. P. Babkin. (Zavodskaya Laboratoriya, 1949, vol. 15, Sept., pp. 1130–1131). [In Russian]. A number of the published spot methods for the detection in various types of steels of chromium, nickel, vanadium, molybdenum, and tungsten.—S. K.

**Spot Tests for Steels.** F. de Ibarra. (Instituto del Hierro y del Acero, 1949, vol. 2, Jan–Mar., pp. 29–32). [In Spanish]. The author describes tests for the semi-quantitative identification of molybdenum and nickel in steels, using the spot technique recommended by Thrun and Heidbrink, by Weirich, and by Schmertner. Complete details are given of the procedure, possible results, time required, and approximate costs. R. S.

**Determination of Small Quantities of Vanadium in Steel and Pig Iron.** B. A. Generozov and N. K. Kuskova. (Zhurnal Anal. Khim., 1946, vol. 1, pp. 325–329 : Chemical Abstracts, 1949, vol. 43, July 25, cols. 5335–5336).

**Colorimetric Determination of Small Quantities of Aluminium in Steel.** N. K. Kyskova. (Zhurnal Anal. Khim., 1947, vol. 2, pp. 7–16 : Chemical Abstracts, 1949, vol. 43, July 25, col. 5328). This method is based on extracting aluminium quinolate with iso-AmOH and determining aluminium in the extract by comparison with standards. Dissolve a 2-g. sample of steel in 24 ml. of 3·6 $N$ $H_2SO_4$. Filter off residue (carbides), fuse with $K_2S_2O_7$ and leach the fusion product with water. Evaporate the solution to 80 ml. and precipitate aluminium, iron, and chromium with $NH_4$ benzoate. If the chromium content is $\geq$ 0·2% reprecipitate. Ash the precipitate and remove silicon with HF. Fuse the residue with $H_2O_2$, cool to 6° and precipitate iron and titanium with a 6% cupferron solution and filter. Evaporate the filtrate to 25 ml., decomposing cupferron in solution, transfer to a 100-ml. metric flask and dilute to the mark. Transfer 10 ml. to a Nessler tube, neutralize solution with $NH_4OH$, acidify slightly with $H_2SO_4$ (litmus paper), and add 4–12 drops in excess, depending on the chromium content. Cool, add 2 ml. of 50% $NH_4OAc$ solution, mix, add 2 ml. of 0·5% 8-hydroxyquinoline solution and mix. Add 10 ml. of iso-AmOH, mix, and compare the colour with standards.

**Instantaneous Spectrography.** J. Orsag. (Société Française de Métallurgie, Oct. 20, 1948 : Revue de Métallurgie, Mémoires, 1949, vol. 46, Dec., pp. 803–805). In quantitative spectrographic analysis the photographic plate has two main disadvantages : The range of linear proportionality between the intensity of illumination and the density of film produced is narrow, and the processing of the plate after exposure takes time. To avoid this the author receives the spectrographic line to be recorded on an electron multiplier. This consists of a photo-electric cell in which the photo-sensitive film consists of cesium-antimony on oxidized silver ; the electron multiplier proper in the same cell, consisting of several grids of magnesium–silver alloy, is very powerful and perfectly linear. By suitable amplification a reading can be obtained on a potentiometer. Several examples of the results obtained by this apparatus are given.—A. E. C.

**Microanalysis of Gases Evolved from Metals.** G. W. Keilholtz and Martha J. Bergin. (Instruments, 1949, vol. 22, pp. 320–321, 360–361 : Chemical Abstracts, 1949, vol. 43, Aug. 10, col. 5646). In the vacuum-tube industry it is important to know the extent to which gases may be evolved from metal parts. A gas-analysis unit was devised in which the gas mixture obtained by heating metals in a vacuum to high temperatures can be collected and analysed by conventional methods. The apparatus is explained and shown in line drawings.

**Determination of Oxygen in Metals by the Vacuum Fusion Method.** R. K. McGeary, J. K. Stanley, and T. D. Yensen. (American Society for Metals, Oct. 1949, Preprint No. 10). An improved type of vacuum fusion apparatus for the determination of total oxygen in metals is described and its operation explained. With the apparatus samples weighing from about 0·1 to 2 g. can be analysed in 15 to 20 min. with an accuracy of ± 0·001%. The apparatus can be modified for the determination of hydrogen and, with the addition of a Toepler pump, can be used for the determination of nitrogen.

**Determination of Nitrogen in Steel.** J. L. Hague, R. A. Paulson, and H. A. Bright. (Journal of Research of the National Bureau of Standards, 1949, vol. 43, Sept., pp. 201–207). A rapid semimicro method for the determination of nitrogen in steel is described. A 0·5–1·0-g. sample is usually required, and the nitrogen is put into solution as ammonium sulphate by suitable treatment with $H_2SO_4$. The ammonia is separated from the bulk of the sample by steam distillation from an alkaline solution. The distillate is collected in boric acid solution, and the nitrogen is determined by titration with 0·01 *N* acid.—R. A. R.

**Contribution to the Identification of Hard Metals (Carbides).** M. Petrdlík. (Hutnické Listy, 1949, vol. 4, June, pp. 165–168). [In Czech]. Individual types of carbides can be identified by physical, electro-analytical, and chemical methods. Of the physical methods, those based on weight measurement are briefly described. Of the electro-analytical methods the Glazunov electrographic method and the one based on anodic dissolution of titanium carbide are described. Details are also given of two colorimetric methods using the peroxide reaction for determining the titanium carbide and the thiocyanate reaction for determination of the cobalt. The solutions for colorimetric analysis are prepared either by dissolving pulverized hard metals or by attacking the surface with suitable agents. Surface attack by drops of acid can be applied for analysis of hard metals which are ready for use. The author uses the Glazunov electrographic method or anodic dissolution of titanium carbide by which titanium is transferred into the electrolyte. The paper deals only with carbides containing WC, TiC, TaC, and cobalt. The practical application of the individual methods is described in detail.—E. G.

**Colorimetric Methods for the Analysis of Electroplating Baths.** D. G. Foulke. (Metal Finishing, 1949, vol. 47, Oct., pp. 58–63, 67). Details are given of colorimetric procedures for determining nickel and cobalt in nickel-plating baths, as well as for determining traces of lead, iron, manganese, copper, cadmium, silicon, and aluminium.—R. A. R.

**Volumetric Determination of the Hydroxyquinoline Compound of Iron.** S. T. Baliuk and V. M. Mirakyan. (Zavodskaya Laboratoriya, 1949, vol. 15, Nov., p. 1368). [In Russian]. A method based on the titration with a standard solution of trivalent titanium in the presence of potassium thiocyanate of the hydroxyquinoline compound of iron is described; it has been successfully used for the determination of ferric oxide and alumina in Dinas, fireclay, dolomite, magnesite, and various clays.—S. K.

**Method for the Determination of Total Sulphur in Ferrous and Non-Ferrous Ores.** A. T. Chernyi and K. V. Podoinikova. (Zavodskaya Laboratoriya, 1949, vol. 15, Sept., pp. 1132–1133). [In Russian]. The determination of total sulphur in ores is described. The sample (0·2 g.) is fused with 3 g. of oxalic acid and 1 g. of metallic calcium at 750–800° C. in a stream of carbon dioxide, the hydrogen sulphide quantitatively produced being absorbed and determined iodimetrically.—S. K.

**Photometric Determination of Sulfur in Metals and Alloys.** C. L. Luke. (Analytical Chemistry, 1949, vol. 21, Nov., pp. 1369–1373). The method described consists of solution of the sample in a mixture of HCl and $HNO_3$, destruction of the $HNO_3$, reduction of the sulphate to sulphide with the aid of hydriodic acid, distillation of the hydrogen sulphide into a solution of ammonium hydroxide, and finally, precipitation as colloidal lead sulphide and measurement of the transmittancy of the sulfide sol at 370 mμ.—R. A. R.

**The Rapid Determination of the Forms of Sulphur in Coal.** (British Coke Research Association, July, 1949, Panel No. 1, Third Report to the Research Committee). The British Standard (B.S. 1016) methods for determining sulphate and pyritic sulphur in coal require digestion in acid for 40 and 24 hr. respectively. Methods are now proposed in which the digestion time is reduced to 30 min. for each acid, and they ensure that the hydrochloric acid will not attack pyrites and that nitric acid will not attack the organic sulphur.

R. A. R.

**Accelerating the Analysis of Sinter.** Iu. I. Usatenko and P. A. Bulakhova. (Zavodskaya Laboratoriya, 1949, vol. 15, Sept., pp. 1128–1130). [In Russian]. Details are given of rapid methods for the determination of silica, iron, and calcium in sinter; they are based on the fusion at 1000° C. of separate 0·5-g. portions of the finely powdered sample with 0·3-g. portions of anhydrous sodium carbonate to give products easily soluble in hydrochloric acid. For the determination of manganese, 0·2 g. of the finely powdered sample is dissolved by boiling in 10 ml. of a mixture of 70 ml. of phosphoric acid with 30 ml. of sulphuric acid. The satisfactory accuracy of these rapid methods was established in numerous tests with samples of sinters and standard samples of ores.—S. K.

**Improved Methods for the Quantitative Analysis of Coal Ash and Coke Ash.** (Department of Scientific and Industrial Research, Fuel Research, 1949, Survey Paper No. 50: H.M. Stationery Office). This paper embodies many alterations found to be desirable as a result of 15 years' further experience in the Coal Survey Laboratories and the Fuel Research Station with the methods published in Fuel Research Survey Paper No. 28 in 1933.

**The Malecki Carbon Monoxide (CO) Detector.** M. G. Amos and A. P. G. Amos. (Instrument Practice, 1949, vol. 4, Nov., pp. 18–21). A portable apparatus for detecting and indicating the concentration of carbon monoxide in the atmosphere is described. The air to be tested is pumped through a drying chamber and two other chambers, the second containing a material similar to hopcalite but inactive, and the third containing active hopcalite. The second and third chambers contain 'Thermistor' resistors connected to opposing arms of a bridge circuit. Any carbon monoxide present reacts with the active hopcalite, generating heat which unbalances the circuit by an amount registered by a galvanometer.

R. A. R.

**Determination of Phosphorus in Ferrochromium.** M. V. Babaev. (Zavodskaya Laboratoriya, 1949, vol. 15, Sept., pp. 1108–1114). [In Russian]. An account is given of a comprehensive series of experiments carried out to test and compare various methods of determining phosphorus in high- and low-carbon ferrochromium. These experiments revealed the unsatisfactory nature of several of the standard methods used in the U.S.S.R., and an improved method is proposed. This was used to determine the distribution of phosphorus in ingots of ferrochromium. The distribution was found to be appreciably uneven in only 10 out of 140 ingots tested, and it is concluded that unsatisfactory analytical methods rather than ingot heterogeneity are responsible for variations in the results of phosphorus determinations.—S. K.

**Influence of Complex Formation on the Magnitude of the Potential of Systems Having Analytical Importance.** V. S. Syrokomskii and E. V. Silaeva. (Zavodskaya Laboratoriya, 1949, vol. 15, Sept., pp. 1015–1019). [In Russian]. In this communication, the fifth of a series on analytical problems, a description is given of the application of ammonium sulphate in acetic acid for the determination of titanium in ferrotitanium and a titaniferous ore. This solution raises the oxidation-reduction potential of the system $Ti^{+4}/Ti^{+3}$ sufficiently to prevent aerial oxidation. Cacotheline is used as an indicator and the titration of titanium can be carried out in the presence of iron. A method is also described for the rapid determination of titanium in quantities of 0·2–5 mg. which gave satisfactory results in the analysis of standard samples of nickel steel containing about 0·4% of this element.—S. K.

**Spectral Methods for the Analysis of Ferro-Alloys.** M. V. Babaev. (Zavodskaya Laboratoriya, 1949, vol. 15, Oct., pp. 1193–1197). [In Russian]. The tendency to segregation of elements in ferro-alloy ingots is discussed, and methods of taking samples from the furnace, from the stream during tapping, or from the solid ingot are considered. Various methods of shaping and polishing electrodes from such samples for spectrum analysis are described and examples are given of the application of the recommended technique to the determination of silicon in carbon-free or low-carbon ferrochromium containing 0·2–3·3% of silicon, and in high-silicon ferrochromium containing 4–18% of this element. The relative error was found to be ±(4–10)% in the first case and ± 10% in the second.—S. K.

**Development of the Spectrum Analysis of Metals.** A. Pokorný. (Hutnické Listy, 1949, vol. 4, Nov., pp. 352–356; Dec., pp. 391–395). [In Czech]. Developments in spectrum analysis are summarized. The effect of several factors on the results obtained is briefly described, *e.g.*, type of spark, and shape of electrodes. New methods, such as direct photometry and the use of electronic counters, for making spectrum analysis more efficient are also dealt with.—E. G.

**New Arc Electrode for Quantitative Spectrum Analysis.** F. Burriel and A. Rodriguez. (Anales de Fisica y Química, 1949, vol. 45B, Nov., pp. 1395–1404). [In Spanish]. The application of the micro-electrode, which eliminates arc oscillations and avoids the effects of fractional distillation, is described. The precautions to be taken are discussed and tables are given.—R. S.

**Experiments in the Preparation of Crystal Plates for Focusing X-Ray Spectrographs.** A. B. Gilbarg. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., pp. 1482–1486). [In Russian]. After a description of two methods for the orientation of the crystallographic planes of quartz crystals, an account is given of the preparation of crystal plates for use in focusing X-ray spectrographs. It is claimed that plates as thin as 56$\mu$ have been prepared by the technique described.—S. K.

**The Influence of Size and Shape of the Specimen on the Results of the Spectrum Analysis of Steels.** V. G. Koritskii and T. B. Edneral. (Zavodskaya Laboratoriya, 1948, vol. 14, May, pp. 558–565). [In Russian]. The effect of size and shape of specimen on the spectroscopic determination of the common elements in steel is dealt with.—S. K.

**The Spectro-Analytical Method of Determining Carbon in Steels and Cast Irons.** K. A. Sukhenko and N. P. Yakovleva. (Zavodskaya Laboratoriya, 1948, vol. 14, May, pp. 625–627). [In Russian]. Some factors entering into the determination of carbon by spectrum analysis are considered and results which have been obtained by this method are examined as regards their accuracy and precision.—S. K.

**Determination of the Carbon Monoxide Content of Air by Combustion.** A. G. Bogdanchenko. (Zavodskaya Laboratoriya, 1948, vol. 14, May, pp. 623–624). [In Russian]. A rapid method of determining carbon monoxide in the atmosphere is described, in which the former is oxidized by the oxygen of the latter by passing through a thin porcelain tube at 800–900° C., the carbon dioxide formed being absorbed in standard baryta.—S. K.

**A New Variation of the Thiocyanate Method for the Determination of Molybdenum.** L. B. Ginzburg and Iu. Iu. Lure. (Zavodskaya Laboratoriya, 1948, vol. 14, May, pp. 538–545). [In Russian]. The use of potassium iodide as the reducing agent in the determination of molybdenum colorimetrically is described.—S. K.

**Drying Samples of Iron Ores at High Temperatures.** N. S. Tkachenko and S. M. Khripach. (Zavodskaya Laboratoriya, 1948, vol. 14, May, p. 625). [In Russian]. A brief account is given of experiments in which it was shown that 100-mesh samples of various iron ores could be completely dried in 5 min. at 150–160° C.—S. K.

**The Use of the Steeloscope for the Analysis of Steam-Turbine Parts.** V. I. Volkov. (Zavodskaya Laboratoriya, 1948, vol. 14, May, p. 627). [In Russian]. The way in which a steeloscope was used for the analysis of inaccessible parts of assembled steam turbines is briefly described.—S. K.

**Rapid Method for the Determination of Small Quantities of Cobalt in a Nickel Electrolyte and in Cathodic Nickel.** M. N. Chepik. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., p. 1470). [In Russian]. In the method described, small quantities of cobalt, in the presence of a large excess of nickel, are satisfactorily determined by potentiometric titration with standardized dilute potassium ferrocyanide, using platinum electrodes.—S. K.

**On Photocolorimetric Analysis.** E. I. Fogelson. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., p. 1469). [In Russian]. The author deplores the continuing failure of the instrument industry of the U.S.S.R. to produce a simple and reliable photocolorimeter in quantities sufficient for the desirable extension of the industrial applications of photocolorimetric methods.—S. K.

**Alkalimetric Method for the Determination of Magnesium Oxide in Calcined Magnesia.** E. I. Dundur and B. A. Bezhan. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., p. 1471). [In Russian]. For the determination of magnesium oxide in calcined magnesia, an alkalimetric method is proposed in which calcium is first precipitated by treatment with sulphuric acid, and iron and aluminium are precipitated by neutralizing with potassium carbonate. Results are shown to agree well with those of the more tedious phosphate gravimetric method. S. K.

**Precipitation of Manganese during the Complete Analysis of Manganese Ore.** V. A. Semenko. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., p. 1472). [In Russian]. A satisfactory method is described for the separation of manganese before the determination of calcium and magnesium in the complete analysis of manganese ore. The manganese is precipitated by titration with potassium permanganate in neutral or slightly alkaline solution, the precipitate being washed with a mixture of 1% nitric acid with 4·5% ammonium nitrate solution followed by 5% ammonium nitrate solution.—S. K.

**Rapid Methods for the Determination of Calcium in Siderites and Sideritic Dolomites.** E. M. Levit. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., pp. 1473–1474). [In Russian]. Two rapid oxalate-permanganate methods for the determination of calcium in siderites and sideritic dolomites are described; they are applicable for concentrations of this element of more and less than 1%, respectively.—S. K.

**Determination of Small Concentrations of Calcium in Steels by the Spectrum Method.** A. G. Komarovskii. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., pp. 1435–1437). [In Russian]. A quantitative spectrographic method is described for the determination of calcium present in steels in concentrations of the order of hundredths of 1%. For making the standard samples used in the production of the working curves, a new method was used which did not require the chemical analysis of the samples. It was found that small concentrations of calcium could be determined both by photometric interpolation and by photographic photometry. The method was tested with calcium-containing samples of steels with approximately 0·3% of carbon, 0·3% of silicon, 0·5% of manganese, and 0·02% each of phosphorus and sulphur, the relative error being approximately $\pm$ 5%.—S. K.

**Photocolorimetric Determination of Cobalt in Iron–Nickel Ores Using Nitroso-P Salt.** D. P. Shcherbov. (Zavodskaya Laboratoriya, 1949, vol. 15, Dec., pp. 1399–1406). [In Russian]. A photocolorimetric method, based on the use of P-nitroso salt, is described for the determination of cobalt present in concentrations of 0·001 to 0·5% in ores containing up to 70% and 5% of iron and nickel, respectively. As regards sensitivity, simplicity, and speed, the method compares favourably with ordinary visual colorimetric procedure, and the range of cobalt concentrations with which it can deal is capable of extension. Precipitated ferric oxide interferes with the determination of cobalt by adsorbing the nitroso salt, and the amount of this salt used must, therefore, be increased when dealing with iron-rich ores.—S. K.

**Spectrographic Determination of Silicon in Chilled Iron.** Z. Karlinski and J. Czakow. (Prace Badawcze Glownego Instytutu Metalurgii i Odlewnictwa, 1949, No. 2, pp. 139–145). [In Polish]. A new quick spectrographic method for the determination of silicon in chilled cast iron containing 0·4 to 2·0% of silicon has been developed. The time taken is about 13 min. and the accuracy is within $\pm$ 2·2%. The samples are 5 mm. in dia. and 70 mm. in length.—W. J. W.

**Polarographic Studies. IX—The Determination of Traces of Metallic Elements (Pb, Cd, Zn, Ni, Co, and Mn), With a Compensating Circuit—Application of the Multi-Capillary Dropping Electrode.** R. Portillo and P. Sanz Pedrero. (Anales de Fisica y Química, 1949, vol. 45B, Nov., pp. 1333–1336). [In Spanish]. In previous parts the authors have indicated the limit of detection of traces of metallic elements, when using a compensating circuit to overcome residual current, and without the necessity of eliminating oxygen contained in the solution. These results showed that the values obtained were within the range of detection normally assigned to the polarographic method. In order to ascertain whether this limit could be reduced, the authors used a multi-capillary electrode. The increase in cathodic surface increased the diffusion current and the sensitivity of the method.—R. S.

**Rapid Analysis of Open-Hearth and Blast-Furnace Slags.** I. Sajó. (Bányászati és Kohászati Lapok, 1950, vol. 5, Jan., pp. 68–70). [In Hungarian]. A rapid method of analysis for open-hearth and blast-furnace slags is described. A 2-g.

sample is sufficient. Solidified slag is crushed and passed through a 576-mesh screen to exclude steel particles ; this is then reground and passed through a 4900-mesh screen. A suspension of the sample in 1–2 c.c. of water is prepared. The procedures for determining silica, lime, iron, iron oxides, manganese, sulphur, and phosphorus are given. A complete analysis takes about 35 min.—E. G.

**The Quantitative Electro-Deposition of Tin from Chloride Solutions. Part II—Stannous Tin Solutions.** A. J. Lindsey. (Analyst, 1950, vol. 75, Feb., pp. 104–105). The advantages of using hydrochloric acid solutions for the rapid electrolytic analyses of metals have already been set forth, and it was shown in Part I of this paper that losses previously noted by various authors were probably due to re-solution of the tin deposit during the process of washing and disconnection. In Part II, the electrolysis of stannous and mixtures of stannous and stannic tin solutions is studied. Strict adherence to the technique of Sand is shown to be essential, and by observing the precautions described, satisfactory results are obtained. In the range 0·1 to 0·3 g. the recoveries of tin show an average error of $\pm$ 0·05 mg.

**Some Characteristics of the Home Produced *p*H Meter SGV-285.** A. F. Vinogradov. (Zavodskaya Laboratoriya, 1949, vol. 15, Nov., pp. 1379–1382). [In Russian]. The results are given of experiments made to determine the variation with time and temperature of the characteristics of the electrodes used in a *p*H meter made in the U.S.S.R.—S. K.

**Report of a Symposium on Laboratory Lay-Out and Construction.** (Royal Institute of Chemistry, Lectures, Monographs, and Reports, 1949, No. 6). The importance of good working conditions and carefully planned construction and layout of chemical laboratories was emphasized at the symposium on this subject held on April 21, 1948 at which the following papers were presented :

**Laboratory Planning and Furnishing,** by C. L. Prior (pp. 5–11).

**Academic and Teaching Laboratories,** by W. H. Linnell (pp. 12–13).

**Analytical Laboratories,** by J. Haslam (pp. 14–19).

**Research Laboratories,** by F. H. Milner (pp. 20–30).

**Microbiological Laboratories,** by G. Sykes (pp. 31–35).

**Laboratory Planning and the Architect,** by E. D. Miles (pp. 36–37).—R. A. R.

### Corrigendum

With reference to the abstract entitled " Clean Gas " in the January, 1949, *Journal*, p. 56, col. 1, it should be made clear that the article does not describe the Whessoe gas-cleaning plant previously installed, but the new one designed and supplied by The International Furnace Equipment Co., Ltd., Aldridge, Staffs.

## HISTORICAL

**An Historical Review of the Progress of the British Iron and Steel Industry.** J. H. Jones. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 11–17). The development of the iron and steel industry is traced, and conditions prevailing during the 1918–1939 period are examined.—J. S. R.

**The Economic Aspect of Sir Henry Sidney's Steelworks at Robertsbridge in Sussex, and Boxhurst in Kent.** H. R. Schubert. (Journal of The Iron and Steel Institute, 1950, vol. 164, Mar., pp. 278–280). Evidence is cited which shows that Sir Henry Sidney with three others was granted a licence by Letter Patent in 1565 to bring up to 100 foreign workers into the country to establish a steel industry. Steel forges were erected at Robertsbridge and Boxhurst in 1565, and were initially successful in competing with steel from Breckerfeld in Germany, but in the period 1568–1571 more and more steel came in from the Swedish Baltic ports and prices fell. Production at the English works appears to have ceased shortly after 1572.—R. A. R.

**The First Iron Handguns in England.** H. R. Schubert. (Journal of The Iron and Steel Institute, 1950, vol. 164, Mar., p. 280). It is shown that small firearms called handguns of brass and of wrought iron formed part of the armoury at the castle of Berwick-on-Tweed in 1389.—R. A. R.

**An Iron Chain of 1252.** H. R. Schubert. (Journal of The Iron and Steel Institute, 1950, vol. 165, May, p. 40). Evidence is furnished by the Liberate Roll of 1252/53 that an order to supply an iron chain to the warder of a polar bear was addressed to the sheriffs of London ; this chain would presumably be of English manufacture.—R. A. R.

**An Early Attempt at Trade-Unionism by Ironworkers in North Derbyshire, in 1814.** H. R. Schubert. (Journal of The Iron and Steel Institute, 1950, vol. 164, Apr., pp. 405–406).

**The Earliest Use of British Cast Iron in Machine Construction.** H. R. Schubert. (Journal of The Iron and Steel Institute, 1950, vol. 164, Apr., p. 406). Evidence is cited from the " forge books " of Robertsbridge in Sussex which shows that hammer hursts were made of cast iron as early as 1554. R. A. R.

**A Rare Group of Wealden Firebacks.** H. R. Schubert. (Journal of The Iron and Steel Institute, 1950, vol. 165, May, pp. 39–40). Three Wealden firebacks (two dated 1589) are illustrated ; these are examples of the transition from the earlier process of manufacture using separate movable stamps to one in which a one-piece model was employed—R.A.R.

## ECONOMICS AND STATISTICS

**The Public Supervision of the Iron and Steel Industry.** (British Iron and Steel Federation, Monthly Statistical Bulletin, 1949, vol. 24, Oct., pp. 1–8). An account is given of the operations performed and experience gained by the Iron and Steel Board which was dissolved on 31st March, 1949, after an existence of two and a half years. The manner in which the supervisory functions carried out by the Board would be fulfilled under the provisions of the Iron and Steel Act are examined.—R. A. R.

**The Structure of the Steel Industry.** Sir Ellis Hunter. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 7–10, 38). The physical set-up of the iron and steel industry is outlined with special reference to the functions of the British Iron and Steel Federation. Some implications of the Act to nationalize the industry are discussed.—J. C. R.

**The Steel Development Plan.** R. Shone. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 39–41, 45). The author reviews the progress made with the steel development plan prepared in 1945 which was a continuation of the process of expanding and modernizing plant initiated before the Second World War. Some of the difficulties encountered are discussed.—J. C. R.

**Fundamental Elements of Steelmaking. Location, Materials and Men.** Sir Walter Benton Jones. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 18–26). In a consideration of the economics of steelmaking, the author discusses the effect of the location of raw materials on steel industries and plants, the labour element, and costs.—J. C. R.

**Steel and Shipbuilding.** Sir John M. Duncanson. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 159–161). The relationship between steel prices and the shipbuilding industry is examined.—J. C. R.

**European Steel Trends in the Setting of the World Market.** (United Nations Department of Economic Affairs : Steel Division, Economic Commission for Europe, 1949, pp. 1–148). The main purpose of this study is to throw light on the probable evolution of European steel production and consumption under present plans, to call attention to some evident dangers, and to indicate the need for intensified European co-operation in this field. It is shown that, if the plans for expansion announced by the European countries are completed, there is a likelihood that production of crude steel in 1953 will exceed requirements by some 8,000,000 tons. Possible remedies are suggested.—R. A. R.

**The Production of Iron in Switzerland.** R. Durrer. (Von Roll Werkzeitung, 1949, vol. 20, Nov., pp. 213–215). The position of the production of iron and steel in Switzerland is briefly reviewed. The electric furnace at Choindez smelting Swiss ore and some smaller electric furnaces on scrap have a combined capacity of 60,000–70,000 tons/annum, but not all of the latter furnaces are operating.—R. A. R.

**Metallurgical Problems of National Importance.** A. Plana Sancho. (Boletin Minero e Industrial, 1949, vol. 28, Aug.–Sept., pp. 377–384). [In Spanish]. Three ideal works, each consisting of coke ovens, blast-furnaces, and steel furnaces, are discussed in relation to theoretical production and yield

from raw materials. The urgent practical problems for the Spanish iron and steel industry are : (1) Better utilization of coal : (2) maximum use of blast-furnace and coke-oven gas ; (3) hot metal charging of open-hearth furnaces ; (4) heat conservation in ingots between stripping and charging to the soaking pits ; (5) continuous rolling-mill development ; and (6) ore screening, mixing, and sintering.

Spain might, with advantage, follow Sweden's example and make sponge iron to relieve the scrap situation. The importance of setting up smaller metallurgical industries in distinct regions of Spain is emphasized. Unorthodox methods should not be overlooked. One of the most important of these is the low-shaft electric furnace. It is important to build new plant to supplement existing plant.—R. S.

## MISCELLANEOUS

**Training for a Metallurgical Career in the Steel Industry. Universities and Technical Colleges.** R. Hay. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 234–237).

**Statistical Methods for Evaluating the Quality of Certain Wrought Steel Products.** E. G. Olds and C. Wells. (American Society for Metals, Oct., 1949, Preprint No. 16). Explanations and examples are given of some of the statistical principles and methods which have proved useful in connection with the evaluation of the quality of wrought steel gun tubes.

**Research and Quality in the Steel Industry.** C. M. Parker. (Iron Age, 1949, vol. 164, Oct. 6, pp. 99–104). The author describes some recent developments in the iron and steel industry, such as coal washing, ore sintering, improved refractories, the use of oxygen, powder scarfing of ingots, electronic controls for rolling mills, and the recovery of manganese from slags.—J. P. S.

**Cooperative Research in the British Iron and Steel Industry.** Sir Charles Goodeve. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 189–197). The author discusses the functions of the British Iron and Steel Research Association in organizing research through its committees and disseminating knowledge through technical conferences. The internal organization of the Association and the fields covered by its research committees are described.—J. C. R.

**Research at the United Steel Companies, Ltd.** F. H. Saniter. (Report of the Conference of Industrial Research Directors and Managers : British Steelmaker, 1949, vol. 15, Oct., pp. 484–488). This brief outline of the Central Research Department of the United Steel Companies, Ltd., deals mainly with the relationship between this department and the management and the other departments.—R. A. R.

**The Use of Oxygen in Metallurgical and Other Thermochemical Processes.** Bo Kalling. (I.V.A., 1948, vol. 19, No. 2, pp. 44–59). [In Swedish]. The principle features of large-scale oxygen-producing plants are outlined. Lachmann has shown it is possible to supply about 40% of the air, uncompressed but chilled, direct to the upper part of the fractionation column, thus greatly increasing the efficiency of the distillation process. The costs of producing oxygen in plants with capacities of 100, 1500, 3000, and 47,000 cu. m./hr. are compared. The transport of liquid oxygen by rail and road is described. The use of oxygen in synthesis gas production, charcoal blast-furnaces, open-hearth furnaces, and Bessemer converters is briefly described.—R. A. R.

**New Resins Provide Practical Bonding Agents for Metals.** E. Preiswerk, K. Meyerhans, and E. Denz. (Materials and Methods, 1949, vol. 30, Oct., pp. 64–66). Particulars are given of the Araldite resins, their applications and properties. They are used for joining similar or dissimilar metals, or metals to non-metallic materials.—R. A. R.

**Land and Maritime Engineering Works at Barceloneta. Description of Plant, Production Units and Main Activities.** (Instituto del Hierro y del Acero, 1949, vol. 2, Jan.–Mar., pp. 39–49). [In Spanish]. These works, founded in 1856 to make boilers, steam and gas engines, and later, locomotives, bridges, cranes, rolling stock. Diesel engines, etc., are described. They include iron and steel foundries, laboratories, and forge, heat-treatment, boiler, and machine shops. The works at San Andres de Palomar contain the most modern plant, including boiler shops, machine, assembly, and general engineering shops where locomotives, tractors, and wheels are produced.—R. S.

**Working Conditions at Certain Melting and Smelting Processes.** W. A. Attwood and W. B. Lawrie. (Fourth Empire Mining and Metallurgical Congress, Great Britain, July, 1949, Paper No. D.6). The paper describes working conditions in blast-furnaces, steelworks, foundries, rolling mills, forges, and glass works. It also describes a method of assessing the effective warmth of an environment where radiant heat is an appreciable factor, and summarizes the principle methods of improving conditions which have been successfully employed in the various industries.—R. A. R.

**Symposium on Health and Safety in the Metallurgical Industries.** (Revue Universelle des Mines, 1949, Series 9, vol. 5, Nov., pp. 378–393). This symposium forms a section of a Congress on Safety, Hygiene and Improving the Appearance of Workshops, held in Belgium in May, 1949 under the auspices of the Belgian Minister of Labour. This section is devoted to the metallurgical industries and after an introduction by P. Haidant the following papers are included :

**Some Particulars of the Muscular Motor,** by A. Govaerts.
**The Organisation of Safety, Hygiene, and Improvement of Factory Appearance in Metallurgical and Metal Processing Industries,** by P. Bougnet.
**Safety Organization in the Iron and Steel Industry,** by Orban.
**Health and Safety in the Zinc Industry,** by A. Pirard.

R. F. F.

**Safety in the Iron and Steel Industry.** E. L. Macklin. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 238–244). Some indications are given of the directions in which safety measures are being adopted in steelworks. Improvements in plant and machinery, increased personal protection, and wider scope of medical services, are some of the main relevant factors.—J. C. R.

**Human Relations in the Steel Industry.** H. Marshall. (Iron and Coal Trades Review, 1949, vol. 159, Sept. 30, Special Issue, pp. 226–233). In considering the human side of the steel industry, the author refers to negotiating machinery, works' magazines, training schemes, canteens, and medical services.—J. C. R.

**Damascene. The Spanish Art of Inlaying Steel with Gold.** F. Valcarcel. (Metalurgia y Electricidad, 1949, vol. 13, Oct., pp. 39–40). [In Spanish]. In spite of its name, this ' damascening ' has nothing to do with the Hispano-Arab art as regards its method of execution. It originated in Spain in 1865. At first, it was done by a method of stamping and later by a method known as ' knife scratching ' or ' striping.'

Fine ' stripes ' are traced in the steel with a knife sharpened on an oilstone. Pure gold wire is inlaid in this stripe and hammered in with fine steel punches. The article is then blued, the steel remaining black and the gold projecting from the hollowed-out stripe. The filigree work is then chiselled and some parts are burnished to throw up the contrasting colours. Silver alloy can also be used to give a beautiful variation in tone.—R. S.

# BOOK NOTICES

DICKASON, A. " *The Calculation of Sheet Metal Work. For Students and Craftsmen.*" 8vo, pp. viii + 250. Illustrated. London, 1950 : Sir Isaac Pitman and Sons, Ltd. (Price 15*s*.)

The greater part of this work has previously appeared in *Sheet Metal Industries* in the form of articles. It covers as far as possible all the various types of calculation problems met with in sheet metal work, beginning with simple surface areas and leading to the more complex volumes and the determination of dimensions for given capacities. The chapters are as follows : (1) Mensuration ; (2) The Circle ; (3) Volume, or Cubic Capacity ; (4) The Right Cone ; (5) Pyramid and Transformer ; (6) The Sphere ; (7) The Spiral Blade ; (8) Dimensions for Vessels of Given Capacity ; (9) Elbows and Bends ; (10) Oblique Chutes ; (11) The Economical Design of Tinplate Containers ; (12) The Accurate Development of Templates ; (13) Riveted Joints ; and (14) Costing and Estimating. An appendix gives numerous samples of the questions on sheet metal works taken from past examination papers of the Union of Lancashire and Cheshire Institutes, the Northern Counties

Technical Examinations Council, and the City and Guilds of London Institute.

"*Gmelins Handbuch der anorganischen Chemie.*" 8. Auflage. System-Nummer 18. Antimon, Teil B 2. "*Schluss des Elements, Verbindungen bis Antimon und Jod.*" 8vo, pp. 129–496. Clausthal-Zellerfeld, 1949. (Price DM 83.60)

The scale of the new edition of "Gmelin" is indicated by the size of this closely printed volume, which presumably represents about a third of the number of pages to be devoted to such a comparatively minor element as antimony. The first 200 pages are occupied by the account of the physical and chemical properties of the element itself, the rest of the volume dealing with the oxides, hydroxides, and halogen compounds. The subject of the alloys has not yet been reached, and the main metallurgical interest of the present volume lies in the discussions of the electrochemical properties of the element and in the account of analytical (including spectrographic) methods for its estimation. To judge from the number of references to journals not easily accessible, a very thorough search of scientific and technical literature must have been made to discover references to antimony and its compounds.

C. H. DESCH.

PARTRIDGE, J. H. "*Glass-to-Metal Seals.*" 8vo, pp. xii + 238. Illustrated. Sheffield, 1949: The Society of Glass Technology. (Price 35*s.*)

This is a particularly good book and the author is undoubtedly master of his subject, being equally at home on both its practical and theoretical aspects.

In Chapter 1 different types of glass to metal seals are classified under four headings, *viz.*: Matched Seals, Unmatched Seals, Soldered Seals, and Mechanical Joints. Chapter 2 is devoted to the properties of metals suitable for glass-to-metal seals. After enumerating the conditions to be met the author goes on to give a detailed account of the metals used in seals. Chapter 3 deals with the manufacture of metals used for sealing to glass, and Chapter 4 with the type of glass used for sealing to metals. The production of seals (matched and unmatched) made by the direct fusion of glass to metals, of seals made with the aid of intermediate metals, and of glass-to-metal seals made by mechanical methods, is adequately covered by Chapters 5, 6, and 7 respectively. Then follows a chapter detailing the practical methods employed for fusing glass and metal. Chapter 9 deals with the qualitative and quantitative measurement of strain in glass in the vicinity of seals, by photoelastic methods. Chapter 11 is highly but usefully theoretical, and covers the evaluation of the principal stresses in a glass-to-metal seal. It contains exactly the right type of theory to assist a well-educated technician to control production. The diagrams in this chapter are particularly praiseworthy. In Chapter 12 the author considers the composition and properties of oxide films and the mechanism of adhesion between glass and metal in seals. The final chapter deals with glass-to-glass, glass-to-ceramic, and ceramic-to-metal seals.

The volume is clearly printed and the diagrams and photomicrographs are excellent.—J. FERDINAND KAYSER.

RUDDLE, R. W. "*The Solidification of Castings. A Review of the Literature.*" Institute of Metals, Monograph and Report Series No. 7. 8vo, pp. 116. Illustrated. London, 1950: The Institute of Metals. (Price 10*s.* 6*d.*)

This book describes methods for the study of solidification rates, and surveys results obtained by their use. Part I deals with the more empirical approach to the problem, namely, studies on methods of gating and feeding where little or no attempt has been made to measure or analyse the actual progress of freezing in the casting. Part II reviews the more fundamental work, where the rates of heat abstraction and rates of solidification have been measured or calculated. The work described in Part II and the conclusions drawn therefrom are briefly summarized in Part III. An Appendix is included containing data on the thermal properties of metals, alloys, and mould materials.

SMITH, G. G. "*Gas Turbines and Jet Propulsion.*" Fifth Edition. 8vo, pp. 386. Illustrated. London, 1950: Iliff and Sons, Ltd. (Price 21*s.*)

The new edition of this work brings the account of the development of gas turbines up-to-date. It has been completely revised and expanded, the established features dealing with the fundamentals of jet propulsion, the basic principles of thrust by reaction and the historic background are retained. Extended treatment has been given to the subjects of combustion, with afterburning for jet units, and to metallurgical problems which are of primary importance to further development. All types of turbine power units—turbojet, turboprop, and compounded—and their influence on military and civil aircraft are discussed.

New chapters deal with ramjets and pulsejets, rocket propulsion, compounded power units, and gas turbines for road transport. Views of the leading authorities on turbine propulsion are set forth in an Appendix.

In the chapter dealing with metallurgical problems the compositions and properties of some typical alloys used for turbine parts are given.

## NEW PUBLICATIONS

BRITISH STANDARDS INSTITUTION. B.S. 1610: Part 1: 1950. "*Verification of Testing Machines.*" Part 1. "*Methods of Load Verification; and Verification of Tensile and Compression Machines.*" 8vo, pp. 11. London: The Institution. (Price 2*s.*)

BRITISH STANDARDS INSTITUTION. B.S. 1617: 1950. "*Mild Steel Castings of High Magnetic Permeability.*" 8vo, pp. 10. London: The Institution. (Price 2*s.*)

BRITISH STANDARDS INSTITUTION. B.S. 1627: 1950. "*Cold Drawn Low Carbon Steel Tubes for Heat Exchangers and Condensers for the Petroleum Industry.*" 8vo, pp. 7. London: The Institution. (Price 2*s.*)

BRITISH STANDARDS INSTITUTION. B.S. 1628: 1950. "*Cold Drawn Alloy Steel Tubes for Heat Exchangers and Condensers for the Petroleum Industry.*" 8vo, pp. 7. London: The Institution. (Price 2*s.*)

BRITISH STANDARDS INSTITUTION. B.S. 1633: 1950. "*Steel for Land Boilers, Receivers and other Pressure Vessels.*" 8vo, pp. 12. London: The Institution. (Price 2*s.*)

BRITISH STANDARDS INSTITUTION. B.S. 1636: Part 1: 1950. "*Terms Used in Spectrographic Analysis, Part 1: Emission Spectrography.*" 8vo, pp. 11. London: The Institution. (Price 2*s.*)

FAIRHALL, L. T. "*Industrial Toxicology.*" Pp. 483. Baltimore, Md. 1949: Williams and Wilkins. (Price $6)

FISHENDEN, M., and O. A. SAUNDERS, "*An Introduction to Heat Transfer.*" London: Oxford University Press (Geoffrey Cumberlege). (Price 15*s.*)

GROTHE, H. "*Metallhüttenkunde.*" Pp. 220. Illustrated. Heidelberg, 1950: Carl Winter's Universitätsverlag. (Price DM 6.–)

HAMILTON, ALICE, and HARRIET L. HARDY. "*Industrial Toxicology.*" Second edition. Pp. 492. New York: Paul B. Hoeber. (Price $7.50)

LINFORD, A. "*Flow Measurement and Meters.*" Pp. xx + 336. London: E. and F. N. Spon, Ltd.

MUREX WELDING PROCESSES, LTD. "*Electric Arc Welding Manual.*" Volume II. "*Metallurgy.*" 8vo, pp. 182. Illustrated. Waltham Cross: Murex Welding Processes, Ltd.

REINER, M. "*Twelve Lectures on Theoretical Rheology.*" 8vo, pp. 162. Illustrated. Amsterdam, 1949: North-Holland Publishing Co.

SCHNEIDERHÖHN, H. "*Einführung in die Kristallographie.*" 8vo, pp. xvi + 369. Illustrated. With "Abbildungen" pp. 102]. Freiburg im Breisgau: Verlag Karl Alber.

SCHULZE, A. "*Metallische Werkstoffe der Elektrotechnik.*" 8vo, pp. x + 403. Illustrated. Berlin, 1950: Metall-Verlag G.m.b.H. (Price DM 24.–)

SMITH, R. E. "*Machining of Metal.*" Revised edition. La. 8vo, pp. 224. Illustrated. Bloomington, Ill. 1949: McKnight and McKnight. (Price $3.50)

SUDASCH, E. "*Schweisstechnik.*" 8vo, pp. 543. Illustrated. Munich: Carl Hanser Verlag. (Price DM 29.–)

UNITED STEEL COMPANIES LTD. "*Elements of Rolling Practice.*" 8vo, pp. 128. Illustrated. Sheffield, 1949: United Steel Companies Ltd. (Price 10*s.* 6*d.*)

## BASIL W. DONCASTER

BASIL W. DONCASTER, Chairman and Director of Messrs. Daniel Doncaster and Sons Limited, was born in Sheffield in 1885, the third son of Samuel Doncaster who was the first Chairman of the Company formed in 1902. His father was the grandson of Daniel Doncaster, who founded the business in 1778.

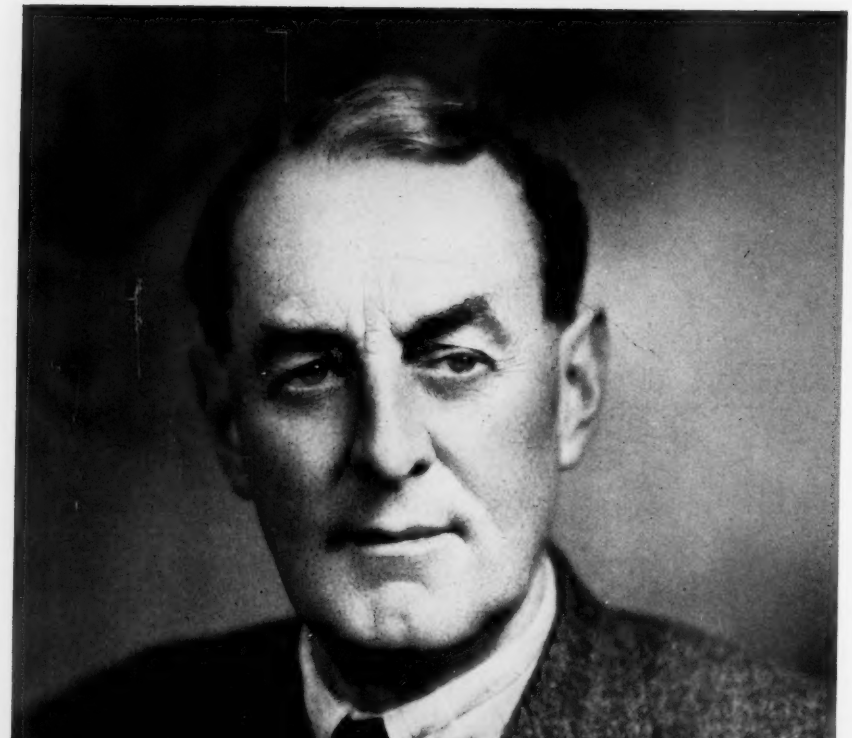
Mr. Doncaster was educated at Leighton Park School, Reading, and at King's College, Cambridge, where he graduated in Mechanical Sciences. On leaving the University he joined his brother, the late Charles M. Doncaster, in the Tool Steel Forge acquired by the Company a few years previously, and actively developed the manufacture and manipulation of crucible steel and Bessemer tool-steel ingots. At this time the Company was largely responsible for the importation and stock-holding of Swedish iron for the tool-steel trade of Sheffield, but later, when the Company began to make forgings, heat-treatment furnaces were installed and thus allowed further developments to take place in the manufacture of alloy steels.

In about 1924 the Company began the manufacture of valve forgings for internal combustion engines and whilst Mr. Doncaster remained in charge of the sales of tool-steel bars he also became responsible for the new Valve Department.

Mr. Doncaster has made numerous trips to Sweden in connection with the import of Swedish materials, and has made other trips to Germany and the United States of America on behalf of the Company. He joined the Board in 1908 and became Chairman in 1948 on the death of his brother.

He lays great emphasis on the importance of personal contact between the directors, executives and workmen, and for many years he has been a member of the works football and cricket teams and is also a keen club cricketer.

Elected a Member of The Iron and Steel Institute in 1924, he became an Honorary Member of Council in 1948 in his position of President of the Sheffield Society of Engineers and Metallurgists. He is Vice-President of the Crucible Steelmakers' Association, and a member of the management committees of the High Speed Steel Association, the Crucible and Tool Steel Association, and the High Speed and Tool Steel Conference.



*Navana Vandyk*

**Basil W. Doncaster**